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American Society for Testing Materials

ORGANIZED IN 1898
INCORPORATED IN 1902

PROCEEDINGS, PART II

The Society is not responsible, as a body, for the statements and opinions advanced in this publication.

SUMMARY OF PROCEEDINGS OF THE ROCHESTER REGIONAL MEETING—SYMPOSIUM ON PLASTICS

The ninth regional meeting of the Society was held in Rochester, N. Y., at the Seneca Hotel, on Wednesday, March 9, 1938, in conjunction with the Spring Group Meeting of A.S.T.M. Committees.

The afternoon session of the meeting comprised a technical program—a Symposium on Plastics. This symposium was sponsored by the Society's Committee D-20 on Plastics. The committee immediately in charge of the technical program consisted of the following:

W. E. Emley (*Chairman*), Chief, Division of Fibrous and Organic Materials, National Bureau of Standards.
H. M. Richardson, Engineer, Plastics Dept., General Electric Co.
J. C. Pitzer, Chemist, The Formica Insulation Co.
L. M. Currie, Director, New Products Division, National Carbon Co., Inc.
H. W. Paine, Chemical Director, Plastics Dept., E. I. du Pont de Nemours and Co., Inc.
Gordon M. Kline, Chief, Organic Plastics Section, National Bureau of Standards.
Louis Shnidman, Laboratory Director, Rochester Gas and Electric Corp. (representing the Rochester Committee on Arrangements).

The purpose of the symposium was to discuss the significant properties of plastics and the various methods available for determining these chemical and physical properties. The symposium consisted of the following six papers:

"The Properties of an Ideal Plastic," by A. F. Randolph, Plastics Dept., E. I. du Pont de Nemours and Co., Inc.
"A Discussion of Testing Methods for the Determination and Comparison of the Strength Properties of Various Organic Plastics," by H. M. Richardson, Engineer, Plastics Dept., General Electric Co.
"A Review of Methods for Measuring the Thermal Properties of Plastic Materials," by W. A. Zinzow, Chief Physicist, Bakelite Corp.

"Flow Relations of Thermoplastic Materials," by C. H. Penning, TENITE Sales, and L. A. W. Meyer, Plastics Research and Development Chemist, Tennessee Eastman Corp.

"Hardness—as Applied in the Plastic Industry," by J. C. Pitzer, Chemist, The Formica Insulation Co.

"Permanence of Plastics," by Gordon M. Kline, Chief, Organic Plastics Section, National Bureau of Standards.

Mr. E. K. Carver, Superintendent, Department of Manufacturing Experiments, Eastman Kodak Co., served as chairman of the session, assisted by Mr. Emley as co-chairman.

The papers, complete with discussion, have been published by the Society as a separate volume entitled, "Symposium on Plastics."

The evening session—the Regional Meeting Dinner—included an address by Dr. Brian O'Brien, Professor of Physiological Optics, University of Rochester, on "Balloon Exploration of the Upper Atmosphere."

Dr. Florus R. Baxter was Toastmaster at the dinner.

There were approximately 450 members and guests in attendance at the sessions.

THE TORSION TEST

EDGAR MARBURG LECTURE[1]

BY ALBERT SAUVEUR[2]

It has been the custom from time immemorial to consider the tension test as the most satisfactory method and to depend chiefly upon its results for the selection of the metals and alloys and their useful applications to our many needs. One would be tempted to infer from this that tensile stresses are the only, or at least the most frequent, stresses to which metallic parts are subjected when in use, and that if we could only guard against failure under tension, all would be well. On reflection, however, it will be realized that failures under simple tension are probably rarer than ruptures under torsional and shear stresses or under a combination of tensile and compressive stresses. Static tensile stresses never cause failure through fatigue, whereas alternate torsional stresses, or repeated twisting stresses do.

Why then have we selected the tension test as the one best suited to our needs? Probably because of its apparent simplicity. To hang increasing weights at the end of a rope until it breaks—is that not the obvious way of ascertaining the strength of the rope?

We realize that faith in the tension test has been so deeply rooted by custom that strenuous efforts will be required to convince the public that the torsion test may be its equal, if not its superior. We are creatures of habit—and we are slaves to our habits. Even those of us who believe themselves to be free thinkers and adventurous spirits hesitate before crossing the line between the known and what they fear to be the unknown, full of pitfalls.

We know in advance that upon being presented with results obtained by torsion testing, the average person will immediately demand that they be translated into tension test figures. This we shall refuse to do. There is no more reason that you as believers in the tension test should insist upon this conversion than we as advocates of the torsion test should demand that your data should be converted into torsional figures. There is no justification here for such arbitrariness. Why should not each method stand on its own? If the torsion test had come into vogue before the tension test, the situation obviously would be reversed. Priority in this case has little significance.

All we can reasonably ask of a testing method is that it enlighten us as to the properties of the materials tested, so that we may use them more intelligently, with greater economy and greater safety. If the torsion test fulfills these requirements as well if not better than the tension test, then it is entitled to our consideration.

After this refusal to have anything to do with the tension test, we relented and conducted a number of parallel tests in the tension and in the torsion machines and found that the torsional strength of the solid bars used by us is about 68 per cent of the true tensile strength, that is, of the strength per square inch not of the original cross-section, but of the reduced cross-section.

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[1] Read on June 29, 1938, before the Annual Meeting of the American Society for Testing Materials, Atlantic City, N. J.

[2] Metallurgical Engineer; Professor Emeritus, Harvard University, Cambridge, Mass.

Having obtained this factor, we ourselves became interested in it because of the consistency of the values obtained with different steels after various treatments, as may be seen by the results shown in Table I.

Our belief that this ratio is really significant finds support in the results reported by Sunatani,[3] as shown in Table II.

TABLE I.—RELATION BETWEEN TORSIONAL STRENGTH AND TRUE TENSILE STRENGTH.

Results obtained by Dr. M. C. Fetzer.

STEEL[a]	RATIO, TORSIONAL STRENGTH TO TRUE TENSILE STRENGTH
18 per cent chromium, 8 per cent nickel, stainless steel	0.65
Ingot iron	0.76
S.A.E. No. 1030 (T)	0.69
S.A.E. No. 1020 (N)	0.66
S.A.E. No. 1010 (N)	0.67
S.A.E. No. 1010 (T)	0.71
S.A.E. No. 1030 (N)	0.67
S.A.E. No. 1030 (A)	0.67
S.A.E. No. 1040 (T)	0.75
S.A.E. No. 1040 (N)	0.66
S.A.E. No. 1095 (N)	0.65

[a] N = normalized; T = quenched and tempered; A = annealed.

It is fully realized that in investigating the torsion test, hollow bars should be used if it is desired to calculate the fiber stresses and to study the relations existing between torsional and tensile properties. Such, however, was not the purpose of our investigations. Our aim was to discover whether the torsion test was not a simpler and more satisfactory method of ascertaining the properties of metals of interest to engineers and other users. For this purpose it is obvious that hollow bars were out of the question. It is, therefore, with our eyes wide open that we used solid bars of standard dimensions.

The Torsion Machine:

As is generally the case with instruments developed in the laboratory, the machine with which our tests were finally conducted resulted from the gradual improvements of previous and less satisfactory types. I shall limit myself to a very brief description of our latest model, as illustrated in Fig. 1.

The main essentials of the machine are the torque wheel *A*, the specimen *B*, and the pendulum *C*. When motor driven the torque is applied by gear *D*.

TABLE II.—RELATION BETWEEN TORSIONAL STRENGTH AND TRUE TENSILE STRENGTH.

Results obtained by Chidô Sunatani.

	RATIO, TORSIONAL STRENGTH TO TRUE TENSILE STRENGTH
Annealed mild steel	0.67
Wrought iron	0.72
Annealed brass	0.65
Annealed copper	0.69

Torque wheel *A*, exactly 8 in. in diameter, is rigidly fastened to the main shaft F_1-F_2. E_1 and E_2 are housings, each of which holds a double ball bearing on which the shaft turns. By using the two bearings E_1 and E_2 bending of the specimen is averted when the load is applied. A $\frac{1}{3}$-hp. 250-v. d.c. motor turns the worm gear *J* which activates gear *D*. Gear *D* is not fastened to the shaft and so in order that gear *D* turn the shaft, it is bolted to plate *G* by two bolts *I*. Plate *G* in turn is keyed to the shaft. For manual loading bolts *I* are removed permitting the shaft to turn freely in gear *D*. The load is applied by hanging weight *K* from the torque wheel. For continuous loading water is run into bucket K at a uniform rate. The torque wheel turns whether the loading is by motor or by hand, and the twist is read in tenths of degrees on Vernier L.

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[3] Chidô Sunatani, "Laws of Failure of Solid Bodies Due to Stress," *Technical Reports*, Tôhoku University (Sendai, Japan), Vol. III, No. 1 (1922).

Blocks M_1, M_2, and N constitute the chucks for holding the specimen; as originally built these chucks accommodated $\frac{1}{2}$-in. diameter bars only. The specimen is held fast by bolting plate N to chuck M_2.

The twisting of the bar turns shaft F_2 which turns on two double ball bearings mounted in housings E_3 and E_4. Rigidly fastened to shaft F_2 is also the pendulum and the recording arms O_1 and O_2. As the load is applied arm O_1 turns with the pendulum, and with the aid of a connecting pin, a toothed bar, and pinion, and suitable guide wheels, needle Q is caused to move so that torque readings may be taken directly from dial P.

One of the features of the machine is the device for obtaining automatic torque-twist diagrams. Suitable graph paper is mounted on drum R which turns freely on its axis. The recording takes place as follows:

1. *Torque.*—Regardless of the amount of twisting in the specimen itself, the applied torque lifts the pendulum a definite amount which causes frame O_2 to move similar to O_1. The turning of frame O_2 causes pinion S to roll in the rack T. Pinion S always remains in a vertical position as its holder V is allowed to pivot freely on frame O_2. Worm gear U which has the recording pen W attached to it by means of a cord, is integral with pinion S. Any movement of gear S pulls the recording pen toward it in proportion to the torque applied or very nearly so. The cord is kept taut by means of the small weight X_1. This weight pulls the pen back in recording yield points and rupture in which cases the rate of torque application becomes negative.

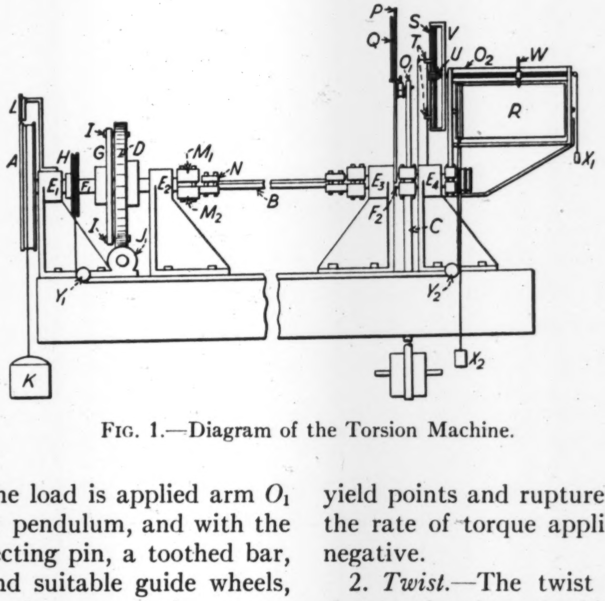

FIG. 1.—Diagram of the Torsion Machine.

2. *Twist.*—The twist is recorded as that turning through which shaft F_1 goes. Wheel H is attached to shaft F_1 with a set screw and as this wheel turns it lets out the cord which twists drum R with the aid of pulley guides Y_1, Y_2, and others which can be seen to good advantage in Fig. 1. Weight X_2 is really the means of turning drum R as it keeps the cord taut when it is released from wheel H.

It should be noted that when using a

constant torque motor to cause the twisting of the test bar, we are conducting the test at a constant twisting speed and observing the corresponding torques. In the tension testing machine likewise we are straining (elongating) at a constant speed and observing the corresponding tensile stresses.

With our torsion machine, however, it was possible, by disconnecting the motor and loading by means of a bucket suspended to a cable wound around the driving pulley, to conduct the test under a constant speed of loading and to observe the corresponding twists. Both lead shot and water fed at a constant speed into the bucket have been employed. As water may be used a drop at a time, there is practically no limit to the slowness with which the stress can be applied. This method has yielded very interesting results.

It was also important to secure, when needed, a very slow speed of twisting, slower than could be obtained by conventional gear boxes; and this was done by using two automobile transmission boxes placed in series.

By this means twisting speed of 360 deg. per hr. could be obtained when both gears were in low (hereafter to be referred to as low low) and 3060 deg. per hr. when both gears were in high (hereafter to be referred to as high high). With both gears in reverse, as slow a twist as 180 deg. per hr. could be obtained.

The need of this very slow speed was made apparent when it was found that under constant twisting speed, the elastic limit was reached very quickly, that is, in a few seconds. Test bars were generally twisted at low low speed to the elastic limit or yield point, and the test completed at high high speed.

The Test Specimen:

After considerable experimenting, a test specimen with a reduced section of 0.350 in. in diameter (Fig. 2.) was adopted as most suitable, although it was found, as was to be expected, that the torsional strength and yield points expressed in inch-pounds were quite independent of the diameter of the test bars.

Two different lengths of reduced section were used, namely, ½ in. and 1 in., and it was found that generous fillets were advisable. Of course, the greater the length of the reduced section, the more marked becomes the proportional limit or yield point.

With very ductile metals the total twist is not a satisfactory measure of the ductility, as it is difficult to obtain check results. Indeed, with very soft metals, such as pure copper, the test bar twists almost indefinitely without breaking. It is believed that for metals which twist more than a full revolution, the stress or torque required to cause a twist of 360 deg. should be taken as a measure of ductility. It is evident that the more ductile the metal, the smaller will be the stress required to twist the bar 360 deg. It is here assumed that we are dealing with test bars of the standard size recommended.

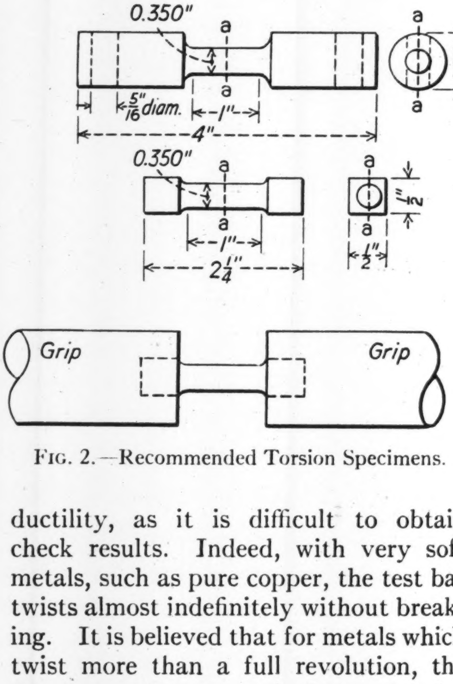

FIG. 2.—Recommended Torsion Specimens.

The grips are of the universal type

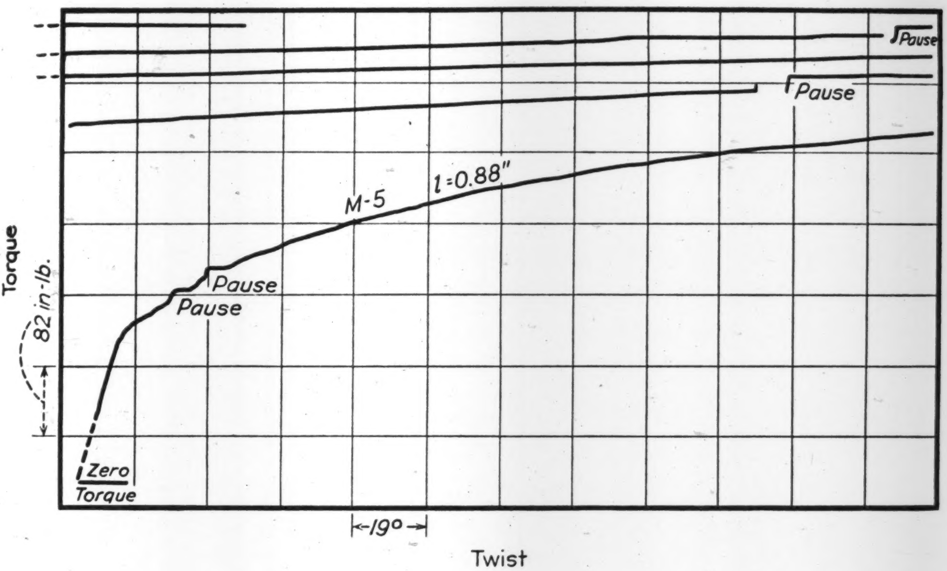

FIG. 3.—Torque-Twist Curve of Normalized Ingot Iron. Water-loaded to first pause.

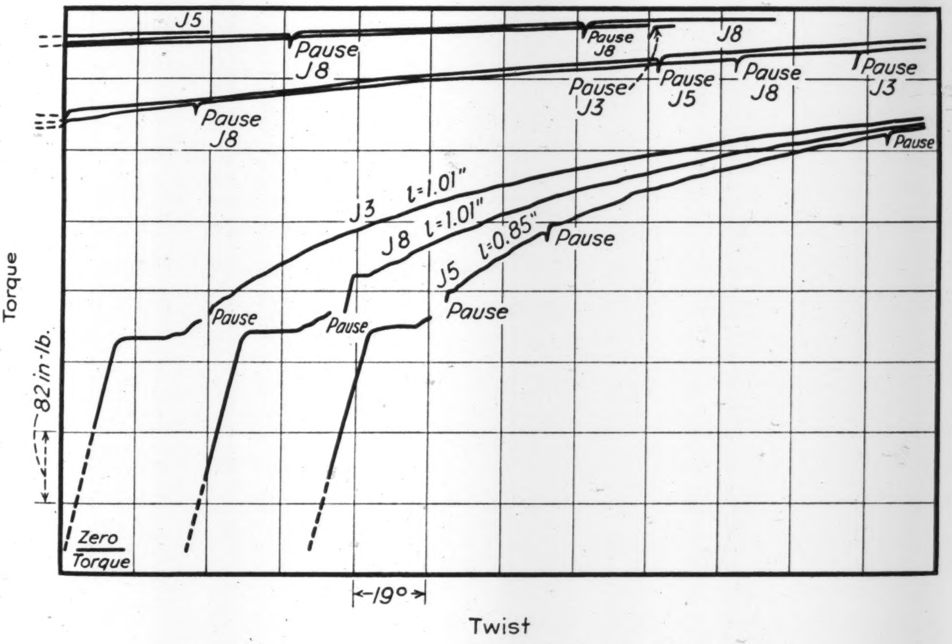

FIG. 4.—Torque-Twist Curves of Normalized S.A.E. No. 1020 Steel. Water-loaded to first pause.

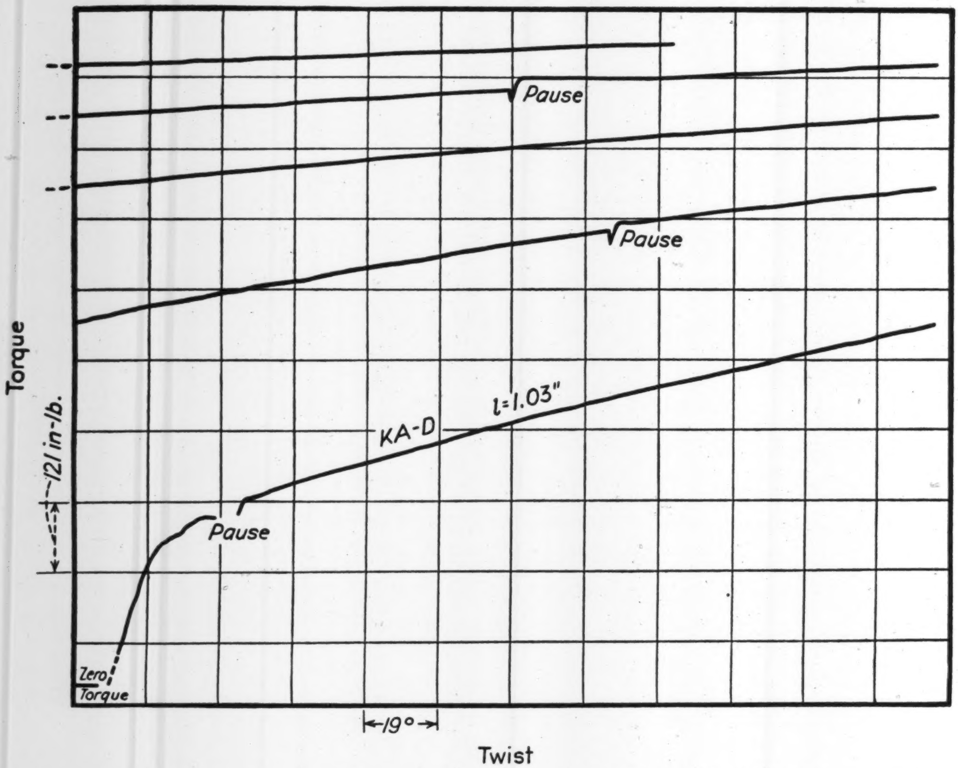

FIG. 5.—Torque-Twist Curve of 18 per cent Chromium, 8 per cent Nickel Stainless Steel. Water-loaded to first pause.

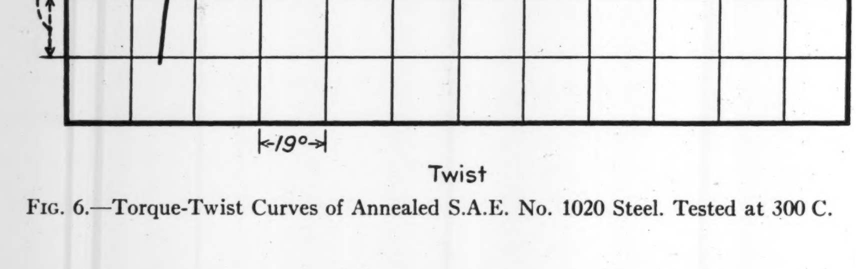

FIG. 6.—Torque-Twist Curves of Annealed S.A.E. No. 1020 Steel. Tested at 300 C.

and capable of accommodating square bars measuring from 0.5 to 1.25 in. or round bars from 0.5 to 1 in. in diameter. In torsion testing it is believed that only standard test bars should be used.

Let us first consider some of the results obtained in testing various steels at room temperature.

Ingot Iron:

Following a time-honored custom we should start with ingot iron. Figure 3 shows the torque-twist curve of normalized ingot iron. When taken we had no means of twisting very slowly by motor driving, hence the test was started by water loading up to the point marked "pause" and then completed by motor driving. The other locations marked "pause" refer to some necessary mechanical readjustments. It will be noted that the yield point is sluggish and that there is no horizontal portion. This was also observed in the tension test.

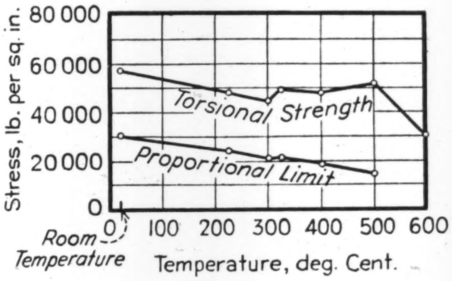

FIG. 7.—Effect of Temperature on the Torsional Properties of Annealed S.A.E. No. 1020 Steel.

A large amount of twist took place before rupture, with comparatively slight increase in stress, which implies little work hardening. In the tension test these features are indicated by a large reduction of area and a small general elongation.

S.A.E. No. 1020 Steel:

A marked yield point is observed followed by considerable twisting (Fig. 4). As the bar twists, however, little increased torque is required which indicates slight work hardening. Three bars were tested and the uniformity of results deserves to be noted, the three curves finally combining into a single one. Better checks could not be expected in tension testing. The figures were:

	YIELD POINT, LB. PER SQ. IN.	TORSIONAL STRENGTH, LB. PER SQ. IN.
J 3	29 000	54 500
J 5	29 700	54 800
J 8	29 800	57 800

Stainless Steel, 18 per cent Chromium, 8 per cent Nickel:

The torque twist is typical of austenite (Fig. 5). The yield point is sluggish, but the steel hardens markedly as it is twisted, as indicated by the increased torque required to continue the twisting. This corresponds to a large reduction of area and to a large elongation in the tension test. The yield point in tension is also sluggish.

Torsion Testing at Elevated Temperatures:

It will be apparent that a torsion machine of this type is well adapted for the testing of metals at elevated temperatures. A suitable furnace placed horizontally on the platform of the machine is all that is needed.

As an illustration, let us consider annealed S.A.E. No. 1020 steel, that is, steel containing 0.20 per cent carbon, tested at different temperatures extending from room temperature to 700 C. The tests were conducted entirely by motor at low low speed to the yield point and completed at high high speed. The curve obtained at 300 C. is shown in Fig. 6. The yield point was 20,600 lb. per sq. in., the torsional strength 44,800 lb. per sq. in. and the total twist 356 deg. The sharpness of the yield point should be noted and the wavy character of the early stage of plastic deformation, a phenomenon to be referred to again a little later.

The results of this series of tests have been plotted as shown in Fig. 7. It will be noted that the blue heat range as indicated by increased strength covers a range of temperature extending from about 325 to 500 C.

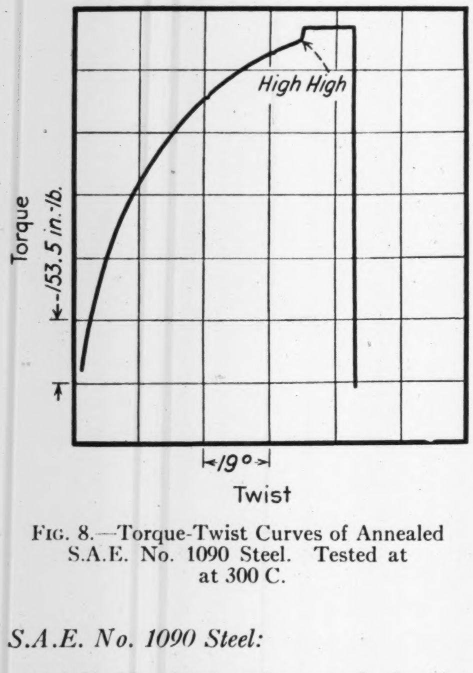

Fig. 8.—Torque-Twist Curves of Annealed S.A.E. No. 1090 Steel. Tested at at 300 C.

S.A.E. No. 1090 Steel:

S.A.E. No. 1090 carbon steel, that is, steel of eutectoid composition, was also tested at different temperatures extending from room temperature to 700 C. It will suffice to show one of the stress-strain autographic diagrams obtained (Fig. 8). This test was performed at 300 C. In a steel of this carbon content there is no detectable yield point. Departure from Hooke's law occurs for a stress of about 30,100 lb. per sq. in. The torsional strength was 75,200 lb. per sq. in. and the total twist, 81 deg. No waviness is observed during the period of elastic deformation.

Twisting in the Blue-Heat Range:

I had occasion as early as 1930 to call attention to the sharp steps exhibited by the twist-torque curves of iron and low-carbon steel when tested in the blue-heat range. It seems advisable to include a brief description of this phenomenon in this lecture in the light of additional results secured since.

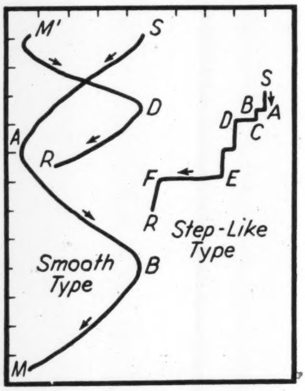

Fig. 9.—Smooth and Step-like Types of Time-Strain Diagrams.

The steps to which I refer are obtained more readily and are of greater magnitude by twisting under constant speed of stressing, which necessitates, as already explained, the use of a bucket suspended by a cable to the driving pulley, while lead shot or water may be employed for loading.

The two types of time-strain curves obtained are shown in Fig. 9; one is smooth, and the other consists of steps. In the step-like type the test started at *S*. From *S* to *A* there was no twist. At *A* there was a sudden twist from *A* to *B* of some 13 deg. From *C* to *D*, covering a period of 21 sec., there was no twist. At *D* there was another sudden twist, and so on. At *E* there was a sudden twist of over 90 deg. and finally fracture at *R*, the total twist being somewhat less than 180 deg. It will be shown that this type of diagram is characteristic of the plastic deformation of ferrite in the blue-heat range. As the load is here proportional to the time, these curves might correctly be described as stress-strain or torque-twist curves.

I ask permission here to quote from a previous paper:

"The plastic deformation of ingot iron in the blue-heat range (Fig. 10) takes place by a series of sudden slips followed by short periods of rest. Starting the test at *S*, the bar had twisted 180 deg. at *A* after 2½ min. through a number of slips showing a tendency to increase in magnitude. At *A* the metal, after a period of rest of some 30 sec. twisted suddenly some 157 deg. From *B* to *C* the twisting was slight and gradual, and the iron finally ruptured at *R* after having undergone another abrupt twist. The total twist was about 1½ revolutions.

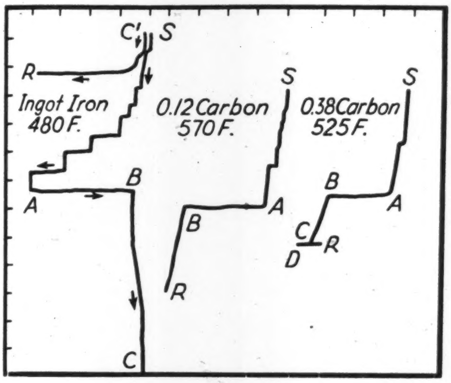

FIG. 10.—Time-Strain Diagrams of Ingot Iron and Hypoeutectoid Steels in the Blue-Heat Range.

"Steel containing 0.12 per cent carbon and, therefore, a considerable amount of free alpha iron undergoes torsional deformation in the blue-heat range in a similar manner; that is, through a number of successive yieldings. We also note that a large twist *AB* of some 120 deg. occurred immediately before rupture took place at *R*. Compared with the behavior of ingot iron, this mild steel does not undergo as many distinct twists, and the total angle is but 180 deg.

"With 0.38 per cent carbon and therefore less free alpha iron, the number of sharp twists is reduced to two, and we again note the occurrence of a pronounced slip *AB* of about 103 deg.

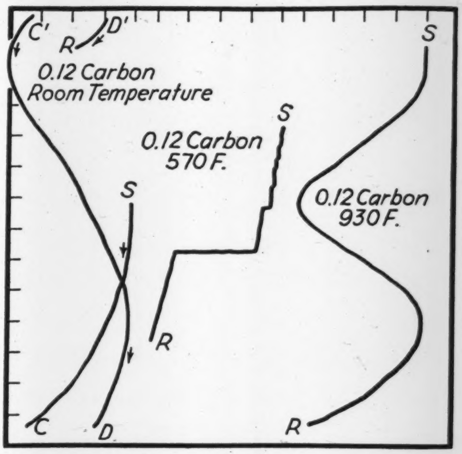

FIG. 11.—Time-Strain Diagrams of 0.12 per cent Carbon Steel, Below, At and Above the Blue-Heat Range.

"In the absence of free alpha iron, the time-strain diagrams of steels tested in the blue-heat range consist of smooth curves, the step-like appearance so characteristic of the curves of iron and hypoeutectoid steels in this range being absent. Even with 0.66 per cent carbon, the curve is free from jerks, although such steel contains some 30 per cent of ferrite. It seems, therefore, that the step-like mode of plastic deformation observed in the blue-heat range of iron and of hypoeutectoid steels must be ascribed to the behavior of alpha iron. It does not follow, however, that eutectoid and hypoeutectoid steels do not undergo a loss of ductility and an increase in strength and hardness in the blue-heat range, although the phenomenon is generally less pronounced, especially in regard to increased strength.

"*0.12 per cent Carbon Steel Below, At and Above the Blue-Heat Range.*—At room temperature the usual smooth curve is observed (Fig. 11). The test started at *S* and rupture took place at *R* after a twist of one turn and 90 deg. At 300 C. (570 F.) the step-like curve characteristic of the blue-heat range is obtained. At 500 C. (930 F.) the curve is again smooth, the steel twisting nearly $1\frac{1}{2}$ revolutions."

An explanation should be sought for the presence of these steps in the blue-heat range and their absence at lower and higher temperatures. This will be done with full realization of its speculative character. As it involves the aging of solid solutions after cold-work deformation, the nature of that phenomenon should be briefly recalled. In order that a solid solution may age, two conditions must be fulfilled: (1) it should possess the urge to age; and (2) it should be given the opportunity. In cold working impure ferrite, considered here as a solid solution, we impart to it an urge to age. At room temperature, however, lack of plasticity may prevent the precipitation of some of the solute, which, accompanied by increased hardness and strength, is generally believed to be the nature of the aging phenomenon. At higher temperature, however, that is, in the blue-heat range, this precipitation may take place because of the opportunity afforded by greater plasticity even if the urge to age resulting from deformation produced at that temperature is less. At higher temperatures, on the contrary, while the opportunity is more favorable still, the urge to age resulting from a deformation necessarily much less severe may be so feeble as not to manifest itself.

With these assumptions before us, let us consider the diagram of Fig. 12, representing the kind of stress-strain curve resulting from testing impure ferrite in the blue-heat range. From *S* to *A* the bar twists elastically, following Hooke's law. At *A* the yield point is reached, and the bar twists quite abruptly through an angle proportional to the distance *AB*. This twist increases its strength for two reasons: (1) because of the cold-work deformation it implies, and (2) because of the precipitation occurring in this cold-worked solid solution. At *B* the bar resists further twisting, while the stress increases from *B* to *C*. At *C* the strength has been sufficiently increased to cause the bar to twist again from *C* to *D*, its strength and hardness being again increased through

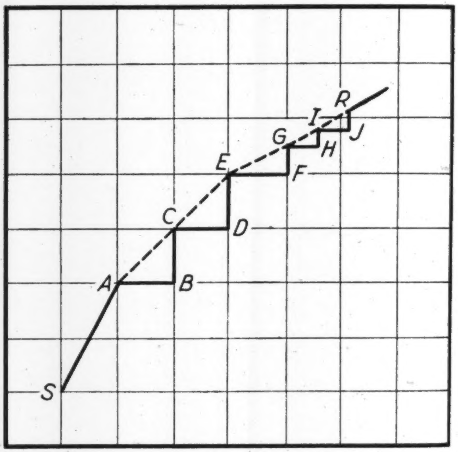

FIG. 12.—Stress-Strain Curve Resulting From Testing Impure Ferrite in the Blue-Heat Range.

deformation and precipitation and requiring now a further increase in stress to cause further twist at *E*, that is, from *E* to *F*, and so on until fracture occurs at *R*.

It will be seen that these successive abrupt twists may be considered as so many yield points, each yield point requiring a greater stress than its predecessor because the bar is now stronger and harder, owing to greater deformation and to precipitation. In uniting the steps as shown by the dotted line, a curve results such as would be obtained in the absence of steps, that is, at temperatures lower or higher than the blue-heat range. At such temperatures, as already explained, yielding by steps does not occur, because of the absence of the two needed conditions to produce them, namely, sufficient urge to age *combined* with sufficient opportunity. I am offering this explanation for what it may be worth, fully aware of its many weak points. I can readily imagine the questions which might be asked if this talk were to be followed by cross-examination, as is usual. This fortunately is one of the rare occasions when the lecturer feels strongly entrenched against such attacks, and for this I render thanks.

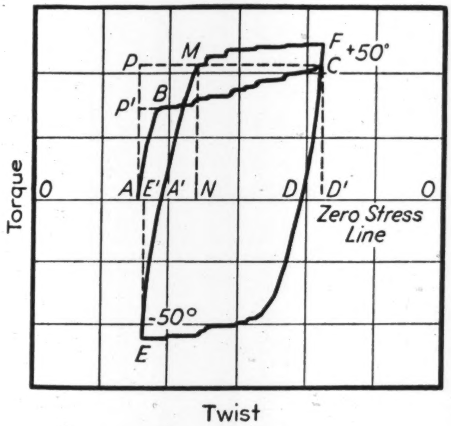

FIG. 13.—Torque-Twist Curves for Ingot Iron.

Reverse Twisting:

Interesting results were obtained by reverse twisting—that is, by twisting the test bar through a certain number of degrees or under a certain torque, followed by twisting in the reverse direction until the same amount of twisting or the same torque has been obtained and then again twisting in the former direction, completing the loop. In the first method we twist in opposite directions until the same angular rotation is obtained; in the latter until the same torque has been applied.

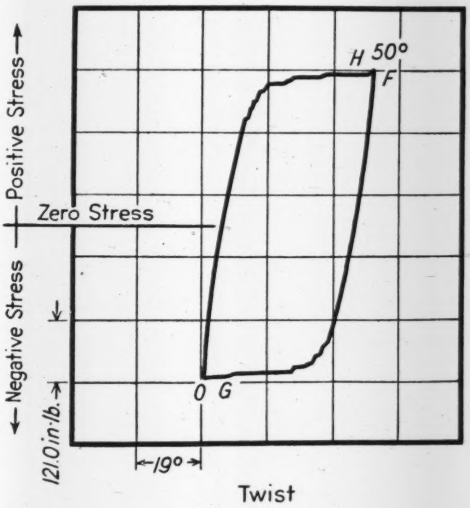

FIG. 14.—Torsional Hysteresis Loop Annealed Ingot Iron.
Second loop on previous bar.

In the diagram shown in Fig. 13, ingot iron was elastically twisted from *A* to its yield point at *B*. It was then plastically twisted to an angle of 50 deg. at *C*, the corresponding stress being represented by the distance $PA = CD'$. The direction of the stress was now reversed and brought back to zero at *D*. The distance *DD'* represents elastic recovery, and the distance *AD* the positive strain or twist remaining in the bar. The stress was now continued to a twist of 50 deg. which may be conveniently designated as −50 deg. The absence of a yield point is due to the fact that we are now dealing with a metal in a cold-worked condition. The torque required to produce this twist of −50 deg. is represented by the distance *EE'* which will be found longer than *PA*.

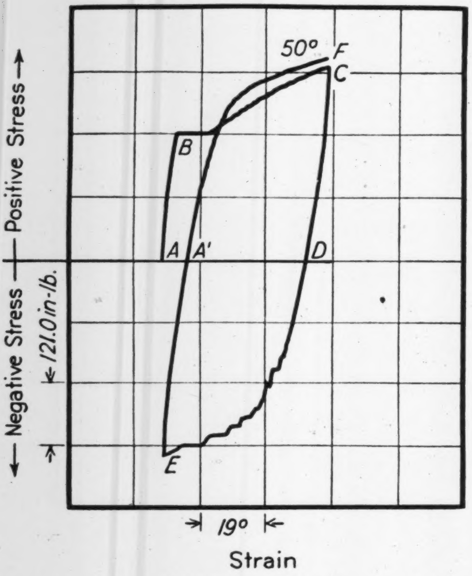

FIG. 15.—Torsional Hysteresis Loop Annealed S.A.E. No. 1020 Steel.

Reverse twist through 50-deg. area of loop = 9.56 sq. in.

In other words, a greater stress is required to produce this −50-deg. twist than was required to produce the +50-deg. twist, obviously because the bar is now in a cold-worked condition. Again reversing the direction of the twist at *E* and continuing it to *F*, when the bar is again twisted +50 deg., it will be noted that at *A'*, that is, for zero stress, the remaining negative strain is represented by the distance *A'D*, elastic recovery having taken place as indicated by *AA'*. At *M* the torque *MN* is equal to the torque *PA*, which in the first positive test had caused a 50-deg. twist. It is now necessary, however, to increase the stress by the amount indicated by the distance *FC*, evidently again because of the cold-work deformation present in the bar. This reverse twisting to a constant twist of 50 deg. in opposite directions was repeated and the diagram of Fig. 14 obtained. The absence of a yield point is due to the fact that we are now testing a cold-worked metal; otherwise the loop is similar to the first one.

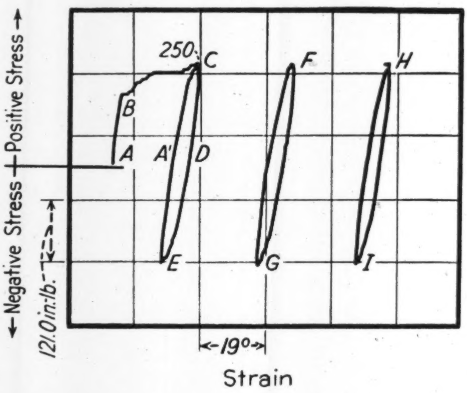

FIG. 16.—Torsional Hysteresis Loop Annealed Ingot Iron.

Reverse twist to a definite stress first in a series of five.

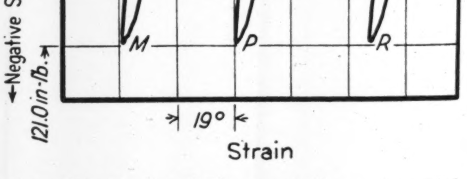

FIG. 17.—Torsional Hysteresis Loop Annealed Ingot Iron.

Second in a series of five.

A test bar of S.A.E. No. 1020 steel was likewise subjected to reverse twisting to plus and minus 50 deg. resulting in the loop shown in Fig. 15. It is larger than the ingot iron loop because it requires a greater torque to produce the same twist.

Testing in reverse directions may be conducted to constant stress as well as to constant degree of twist.

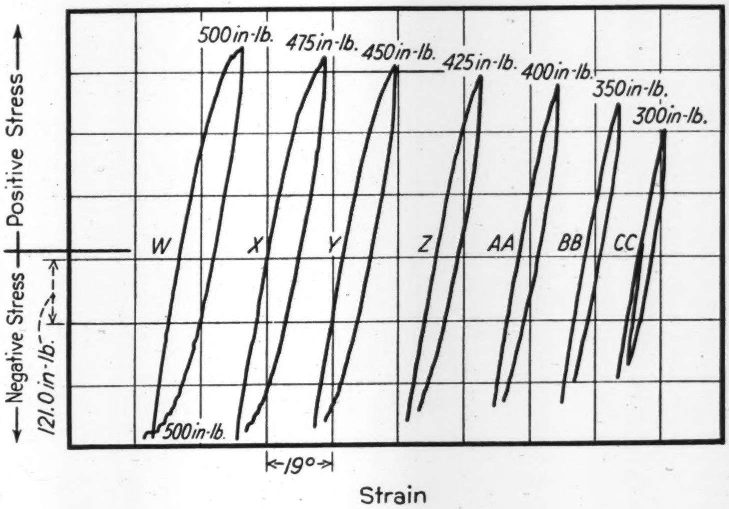

FIG. 18.—Torsional Hysteresis Loop Annealed Ingot Iron. Fourth in a series of five.

One such series of tests will be reported for annealed ingot iron (Fig. 16). The metal yielded elastically from *A* to *B* and plastically from *B* to *C* under a predetermined stress of 250 in-lb. The stress was then released to zero at *D* and then applied in the other direction from *D* to *E*, again released to zero at *A′* and reapplied in the former direction and again to a value of 250 in-lb. It will be noticed that the loop is closed, which signifies that the second application of the stress resulted in exactly the same angle of twist. Two additional loops *FG* and *HI* were obtained, and it will be seen that they are identical.

Similar tests were conducted to a maximum and constant stress of 315 in-lb. (Fig. 17). The loops are now longer because of the greater stress and wider because of the greater twist necessarily resulting from a greater stress. Further increase of stress magnifies these features, the loops increasing in length and in width. As the stress approaches the breaking torsional strength, however, it is found that in order to close the loop, additional twisting at the predetermined stress is needed.

The diagram, Fig. 18, brings out clearly the increase in length and width of the loops as the stress increases.

Comparing a low-carbon, ductile steel with a higher carbon, stronger steel and looping by reverse twisting to constant torque, it is evident that both loops will be of the same length, but that the soft steel loop will be wider because of a greater twist. The area of that loop will likewise be greater. Looping to constant twist on the contrary, while yielding loops of equal width, will

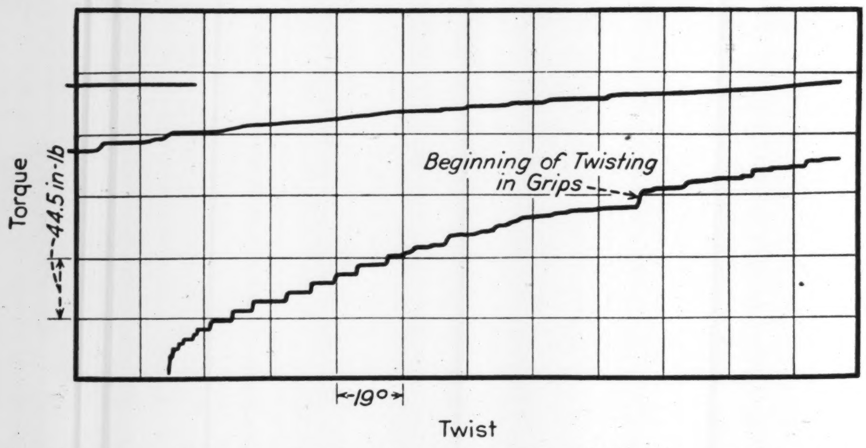

FIG. 19.—Torque-Twist Curves of Pure Copper Single Crystal Test at Room Temperature.

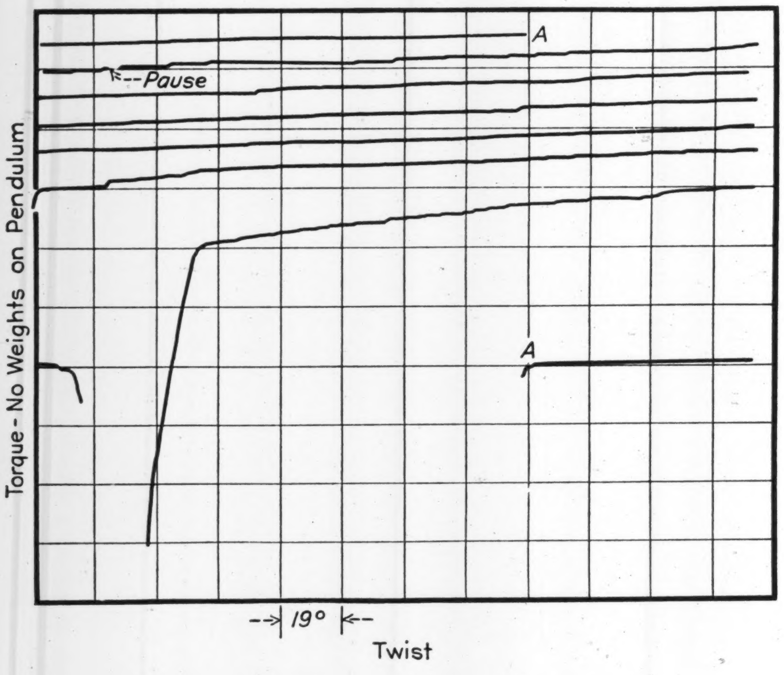

FIG. 20.—Torque-Twist Curves of Pure Copper.
Polycrystalline test at room temperature.

result in a longer loop and greater area for the harder steel, since it will require a greater stress or torque to produce the same twist.

The areas of the loops can readily be determined by planimeter measurements. This has been done for ingot iron and for S.A.E. No. 1020 steel after reversed twisting to an angle of 50 deg.

The area of the ingot loop was 7.37 sq. in. while that of the S.A.E. No. 1020 steel was 9.56 sq. in.

There seems to be little doubt but that useful information could be obtained from a closer study of the significance of these hysteresis loops. Close relations must exist between them and the physical properties of metals which are not readily brought out by the tension test. It also seems to afford an excellent and simple method of studying the effect of cold work deformation at room and at higher temperatures, including the blue-heat range.

Torsion of Single Crystals:

In order to ascertain the behavior of single crystals when subjected to torsion, single crystals of copper of high purity were tested. These measured about 3 in. long and $\frac{1}{2}$ in. in diameter. They were produced by the Bridgman method—that is, by causing crystallization to take place from a single nucleus. The test was conducted throughout at the low low speed, about 1 rev. per hr. The diagram obtained is shown in Fig. 19. The yielding by "steps" or "jumps" should be noticed. After a certain amount of twisting, the metal in the reduced section had been hardened to such a degress that the metal held in the grips began to twist. This made it impossible to record the breaking strength at the reduced section.

The successive strains, that is, the horizontal portions of the steps, have a tendency to increase as the test proceeds, while the increase in stress necessary to cause the next step, that is, the vertical distance, decreases. It is also significant that the steps gradually disappear.

A bar of pure copper, polycrystalline in nature, was tested in a similar manner and the curve shown in Fig. 20, obtained. The yield point is sharply marked, and the steps very much less pronounced.

Speculating, it might be conceived that twisting in the plastic range takes place by block slipping and that in the case of a polycrystalline metal and also in single crystals fragmented by deformation the slipping blocks are so small that the individual steps are not recorded on the curve.

It may be that each step corresponds to the occurrence of one set of slip bands.

The torsion machine used in these investigations has also been applied to the determination of creep at various temperatures by Dr. M. C. Fetzer, and results well worth recording obtained.

It is with regreat that lack of time prevents me from placing them before you. However, a paper by Dr. Fetzer soon to be published will supply those who may be interested with his results and their discussion.

What I claim for the torsion test is: (1) simplicity of manipulation, (2) low cost of torsion testing machine, (3) low cost of preparation of test bars, (4) adaptability to high temperature testing, (5) possibility of reverse twisting, (6) possible adaptability to determination of creep, (7) possibility of testing at will under constant speed of twisting or under constant speed of loading, (8) determination of properties unrevealed by the tension test.

In conclusion I should render unto Caesar that which is Caesar's. The results reported in this lecture have been culled from the theses for the doctorate degree of three men to whom all credit should go, if credit is in order, namely Dr. Ralph Leiter, Metallurgist, Edward G. Budd Manufacturing Co.; Dr. Daniel Eppelsheimer, Metallurgist, Union Carbide and Carbon Research Laboratories; Dr. M. C. Fetzer, Instructor in Metallurgy, The Pennsylvania State College.

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SYMPOSIUM ON IMPACT TESTING

INTRODUCTION

By M. F. Sayre[1] and Walter W. Werring[2]

Service failures of materials often occur with little deformation. They are therefore sudden and often disastrous surprises and have naturally attracted the attention of engineers from quite early times. However, the development of special machines designed to subject materials to impact forces as a means of investigating brittleness began only about 1900. Since then the engineering literature on impact has become of increasing importance, and a considerable variety of test machines and methods have become established in practice. Individual types of testing machines have usually been associated also with a particular design of specimen and this has resulted in much uncorrelated data. Discussion over the relative merits of particular methods of test has been rife. However it has become increasingly evident that liability to impact failure results from the influence of a number of different factors upon the impact properties of materials and in specific cases different combinations of variables may cause very unlike specimens and machines to yield equally valuable and significant data. It is because of recognition of the fact that there is probably no one best machine or specimen for every purpose that the Society has moved slowly in standardization of impact tests. Assuredly it has not been because of any lack of interest in the subject.

Although impact testing of particular types of material has been actively investigated by a number of committees, general discussion of impact problems over a period of years has centered in the Society's Committee E-1 on Methods of Testing. As long ago as 1922 Committee E-1 sponsored an ambitious Symposium on Impact Testing.[3] Interest in the subject has been intensified by the increasing demands made upon materials by modern design requirements. Also the development of the use of welding with its consequent discontinuity of microstructure in metals and its differences in thermal treatments has initiated new and important studies of impact properties and tests.

At the annual meeting in New York in 1937 an informal round-table discussion of impact and impact testing was held under the joint sponsorship of Committee E-1 of the A.S.T.M., and the Americal Welding Society and the Welding Research Committee of the American Institute of Electrical Engineers. The attendance was large and interest continuous. In conclusion it was agreed that a formal symposium under the same auspices should be arranged for the following year to present as far as possible the present state of opinion on the theory and practice of impact testing of homogeneous materials and welds. The present 1938 symposium is the result of this decision.

Because of the great variety of test

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[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 22, Part II, p. 5 (1922).

methods in use, some may find it difficult to recall in detail all of those which may be mentioned. Therefore the following brief descriptions of the usual tests are given:

Proof tests are those in which a specimen or finished part is subjected to a specified blow which it must withstand without failure or with a specified maximum damage. Examples are the "drop tests" for railroad car axles and wheels in which heavy weights are dropped upon the parts. Proof tests are often specified to be applied to 100 per cent of the manufactured product.

Increment drop tests are those in which a weight (usually known as the tup) is dropped upon a specimen from a height which is increased in steps until failure occurs.

Single-blow drop tests are also made by special machines which drop a tup of sufficient weight to break the specimen with a single blow. In these machines various means are provided to determine the excess energy remaining in the tup after fracturing the specimen so that the energy absorbed may be determined by difference from the initial energy. Such machines are the Fremont, the Olsen, and the Hatt-Turner, the latter used extensively in tests of wood. The Fremont specimen is 30 mm. long with a cross-section 8 by 10 mm. A notch 1 mm. square is cut across one of the 10 mm. wide faces of the specimen. It is broken at the velocity of blow of 29 ft. per sec.

The Olsen machine is capable of breaking standard $\frac{1}{2}$-in. diameter threaded tension specimens in impact.

Pendulum impact machines.—A variety of machines using the pendulum principle have been developed. In these the specimen is broken by a single blow of a heavy calibrated pendulum. The energy absorbed in the fracture of the specimen is readily determined by calculation of the energy of blow indicated by the initial position of the pendulum and the remainder indicated by the highest position on the opposite side which is reached by the pendulum after breaking the specimen. Tests on the pendulum machines may differ in the design of the specimen and in the manner in which it is supported and struck. This gives rise to the following well-known tests.

The Charpy test which takes its name from its originator is one in which a notched specimen is supported at the ends and struck in the middle. The specimen is a bar of rectangular cross-section, notched on one side in the middle of its length. The blow is struck on the side opposite the notch at a maximum velocity of 17.4 ft. per sec.

The Charpy notch originally standardized by the International Association for Testing Materials was of the keyhole type made by drilling a hole and then cutting through from one side of the specimen to the hole. The hole is 4 mm. in diameter and the saw cut is 2 mm. wide. The standard specimen is of square section 30 by 30 mm. It is 160 mm. long and is tested on a span of 120 mm. The notch extends half way through the specimen in the direction of the blow. A similar proportionally smaller specimen 10 by 10 mm. in section with the same type notch is recommended for special uses. Still smaller supplementary specimens for the Charpy test on 40-mm. spans are now under consideration abroad with a modification of the classic keyhole notch and also with the sharper 45-deg. V notch which has been found necessary to insure differentiation between tough materials. France, Germany, and Italy have all adopted different notches for supplementary specimens.

The Izod test, of British origin, is also made on a pendulum machine. The specimen is held in a vise with the center line of the notch on the level of the top of the clamping surface. The blow is struck on the notched side at a point 22 mm. (0.866 in.) above the edge of the clamp at a velocity of 11.5 ft. per sec. The standard specimen is 10 by 10 mm. and the notch is a 45-deg. V, 2 mm. (0.079 in.) deep with a radius of 0.25 mm. (0.010 in.) at the bottom. A round specimen with a similarly shaped straight notch is also used to a considerable extent. In the round specimen a flat parallel to the bottom of the notch is milled where the hammer strikes.

The Oxford machine (University of Oxford) is a pendulum machine of new and radical design in which both the specimen anvil and the tup are mounted on a pendulum. Both pendulums are supported only on wires with the intention of minimizing transmission and dissipation of energy through the machine to earth. The motions of both anvil and tup are recorded.

The specimen is loaded in the Oxford test as a beam of constant moment by means of a yoke which applies the force of the blow at two points $1\frac{1}{2}$ in. apart equally spaced from the end supports which are $4\frac{1}{2}$ in. apart. The specimen is $5\frac{1}{2}$ in long

The Oxford specimen is cylindrical and $\frac{23}{32}$ in. in diameter. It has a short section of reduced diameter in the center in which a sharp bottom circumferential 90-deg. V notch is turned.

Flywheel machines use a rotating flywheel as a source of energy for the fracture of the specimen. The knife edge or striking member is held within the rim of the wheel until the desired rotational speed is attained. The striking member is then extended and the specimen is engaged and broken in the next revolution.

The Guillery machine is of the flywheel type arranged to break standard Charpy specimens rigidly supported in the usual manner. The velocity of blow is 29 ft. per sec. The energy of the flywheel both before and after fracturing the specimen is indicated by a manometer reading of the output pressure of a small turbine coupled to the flywheel.

The energy lost by the flywheel in breaking the specimen is comparable to the loss of energy of the pendulum in the Charpy and Izod tests.

The Watertown variable velocity machine is also one in which the energy of blow is generated by rotation of a flywheel and the specimen is struck by horns which are extended after the proper speed is attained. It differs from the Guillery machine in very fundamental ways. The flywheel furnishes the energy for rupture at a range of velocities up to 1000 ft. per sec. but is not involved in the measurement of energy values after the impact. The specimen is mounted upon a pendulum which is normally at rest. When the specimen is struck it imparts an impulse to the pendulum. The motion of the pendulum is indicated by a dial gage reading from which the energy absorbed by the specimen is derived by means of an elaborate mathematical analysis of the machine's energy relations.

In all tests with this machine the specimen is broken in tension. Round specimens with threaded ends are used and are usually $\frac{3}{4}$ in. in diameter and about 3 in. long with a reduced section in the center. The length chosen for the reduced section is dependent upon the extent to which notch effects are desired in the test.

For the purposes of identification each of the foregoing machines and tests have been described in their individual and original form. Because of the widespread use of these different tests some commercial impact testing machines are designed to make Izod, Charpy, and tension impact tests. Both Charpy and Izod type pendulum machines have for many years been equipped to make tension impact tests by threading specimens into the back of the pendulum and arranging to have the free end of the specimen catch on stationary supports as the pendulum swings.

This is an imposing list of differing types of impact machines and of impact specimens, and the variety has tended to increase rather than decrease in present practice. The tendency in many past discussions of impact testing has been to emphasize the study of these different types with a view to finding the field of use of each one, and possibly to standardizing on some one particular type. In the present symposium, this phase of the question has been intentionally suppressed in favor of two other phases which have seemed much more important.

The first of these is a discussion of present fields of commercial use for the impact test with particular reference to places where the impact test gives necessary information not supplied by static tests.

Second is a discussion of the basic theory of the impact test. Inherently, at the present moment, this is much more important and valuable than any discussion of details of testing. It has long been recognized that occasionally slight changes either of temperature, velocity of blow, or shape of piece may transform a normally tough, ductile machine part into a brittle unsafe member. The need now is for a better understanding of the way in which the impact test can be used to measure for any given material or machine part the safety factor against this type of failure.

It is in the interest of promoting more widespread knowledge of these two phases of impact testing that this symposium is presented.

UTILITY AND NON-STANDARD IMPACT TESTING

By Sam Tour[1]

Synopsis

A distinction is made between the "science" and the "art" of impact testing. The science is defined as dealing with the technique of the test and test conditions and evaluation of results. The art is defined as including all forms of impact tests as applied in industry.

A plea is made for a wider use of the impact type of test in industry even though a full theoretical or mathematical analysis and explanation of results cannot be made.

Examples of utility and non-standard impact tests are described for the following materials: fiber board, wood, concrete, stone, enamel, automobiles, tire chains, small rollers, steel spring stock, gear teeth, cap screws, pipe and tubing, thin wall tubing, and rails.

The need for a wider study of impact testing is stressed by the citation of present day problems on non-breakable glass and molded plastics.

Suggestions are made as to the adaptability of standard machines to non-standard and utility impact testing.

Impact testing as a means for detecting the presence of brittleness or the comparative toughness of materials has been practised for generations. Whether the impact blow is produced in an elaborate machine upon a carefully prepared specimen or by the crude method of wielding a hammer by hand upon an irregularly shaped rough-finished piece of material, the purpose of the test is the same: to detect brittleness or relative toughness.

The technical literature contains numerous contributions on the subject of impact testing. In certain specific applications, the "art" has been advanced to a "science." The "science" of impact testing has to do with the details of the test itself and the significance of numerical results of the test. The "science" of impact testing promotes a careful study of the technique used in the test, a careful analysis of the testing conditions used, a standardization of the test specimens, a critical evaluation of the results. These are all very worth while.

This paper is not presented as an argument against the "science" of impact testing. It is presented for the sole purpose of emphasizing the importance of the "art" of impact testing as applied in the form of utility tests of materials and structures. While "science" indicates the need of a standardized test specimen tested on a standardized machine, it should not attempt to exclude the use of non-standard specimens nor the use of non-standard machines. The "art" must be sufficiently practical to permit of its being applied in industry where the presence or absence of excessive "brittleness" is of vital importance. The "science" is not bound by such a limitation.

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[1] Vice-President, Lucius Pitkin, Inc., New York City.

During the past twenty years, we have seen various attempts made to limit the applications of the impact test by indirection. Attempts have been made to prescribe specific sizes and shapes of test specimens, of notches, of striking velocities, of relationships of centers of percussion to centers of gravity, of shape of striking edges, of rigidity of support of machine and specimen, of units and terms used in recording of results. As applied to specific utilization cases, these attempts at standardization are all worth while. As applied to the general "art" of impact testing, they tend to stifle and retard advancement. Thousands of sizes and shapes of articles in daily use must have a minimum degree of toughness which can be found by impact tests even though the articles are not of such shape or size as to permit of being subjected to one of the so-called standard tests.

While "science" debates the stress distribution around notches of various sizes and shapes on test specimens, engineers and architects continue to design machines and structures subject to sudden loading and embodying all possible varieties of notches. The nature of the notch or notch effect is usually determined by unavoidable circumstances or details of the design. As the "science" develops, our knowledge of the sequence of events which lead to failure at a notch is promoted. The actual failure as the climax of this sequence of events is the all-important point to the prospective user of the machine or structure. How can he be reasonably assured as to its safety? The answer may be found by comparison with successful machines or structures of similar design. Lacking such examples or not wishing to depend entirely on such examples, a service test may be made. Service tests, however, often involve many parts assembled into a finished machine or structure where the failure of one part causes damage to many other parts. Due to the excessive cost or great hazard involved in such cases, it is highly desirable to carry out tests on the individual items entering into the assembly. How are these tests to be made?

Are they for the purpose of testing the quality of the material of which the part is made, or the quality of the finished part itself? The "science" of impact testing leads to the belief that the energy absorbed per square inch of break is not a pure function of the area of the break even for similarly proportioned specimens of practically identical materials. We cannot translate the values obtained on a standard Charpy or Izod impact test specimen into design figures for a machine or structural part of different size and shape than the test specimen. The standard test tells something of the quality of the material from which the specimen was machined, but little as to the service characteristics of the part from which it was taken. Some test of the part itself becomes necessary. This test is in fact a utility test. If the part in use is subject to suddenly applied loads, the test involved should use suddenly applied loads. Such a test is a utility test in the field of the "art" of impact testing.

The part itself may not be such as to permit of being subjected as a whole to a utility test. Its size and shape may be such that only a portion of it is used for the test. If the portion so used is not in the shape of a fully machined or formed impact test specimen, we again have a utility impact test. If the portion is fully machined or formed but is not to standard impact test specimen dimensions, we have a non-standard impact test.

The following portion of this paper deals with various examples of utility and non-standard impact tests which have proven of value in industry. The material has been gathered from various sources. An attempt will be made to give due credit to each source. These examples deal with various materials such as fiber, wood, concrete and enamel, as well as with steel.

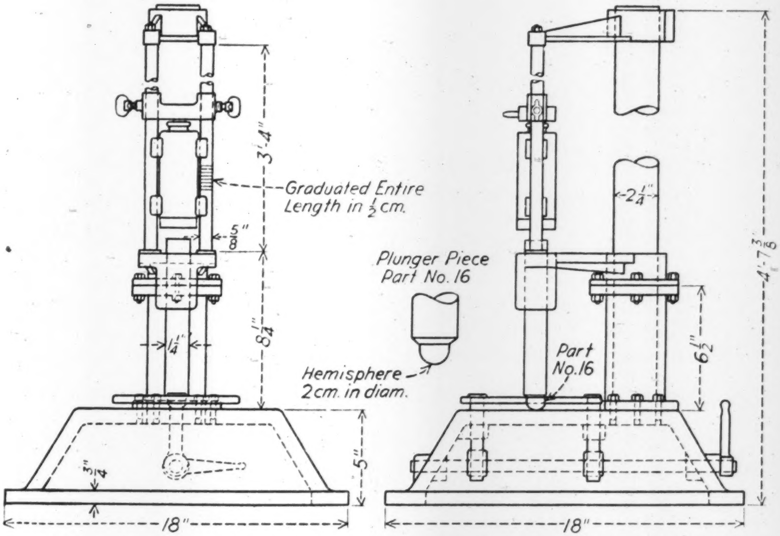

FIG. 1.—Diagram of Impact Tester.

Fiber Board:

T. D. Jarrell, Associate Chemist, Industrial Farm Products Research Division of the United States Department of Agriculture, Bureau of Chemistry and Soils, Washington, D. C., writes as follows:

"During the War some work was done by the Bureau of Chemistry and Soils in cooperation with the Navy Department in the preparation of specifications for solid and corrugated fiber containers for canned goods for overseas shipment.

"In the course of this work an impact tester was developed for determining the resistance to impact of the fiber boards, and an article entitled "Impact Tester for Fiber Board," was published in *Paper*, Vol. 24, No. 21, July 30, 1919, pp. 24–27."

From the article referred to, the following is quoted:

"Since the serviceability of fiber containers, for which fiber board is chiefly used, is so largely dependent on its resistance to impact, the determination of this resistance would seem to offer additional information on serviceability, helpful alike to shippers, box makers and board manufacturers.

"The Bureau of Chemistry has therefore attempted to adapt the *principle of the impact testing machine used for cement and rock* to show more nearly the strength of fiber board under actual service conditions than can now be done by the testers generally employed."

The design of the machine is shown in Fig. 1.

This type of drop impact test machine

has been employed in a number of other industries for a variety of impact tests on various materials. One specific example is its use on vitreous enamel coatings on metals. The increment drop impact test with the Hatt-Turner machine described in A.S.T.M. Standard Methods of Testing Small Clear Specimens of Timber (D 143 - 27)[2] is an example of this type of test as standardized for a particular class of material.

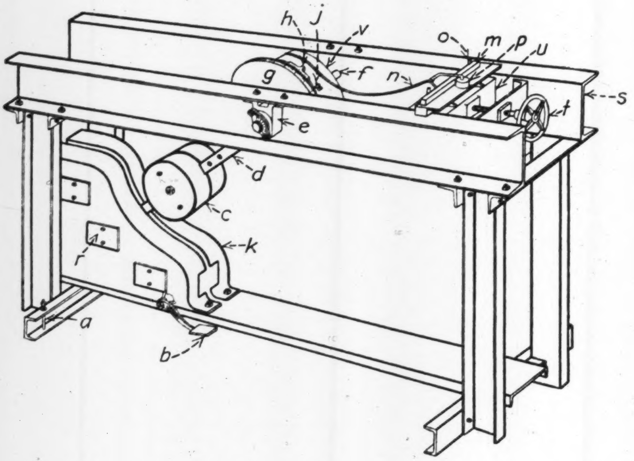

FIG. 2.—Forest Products Laboratory Toughness Testing Machine.

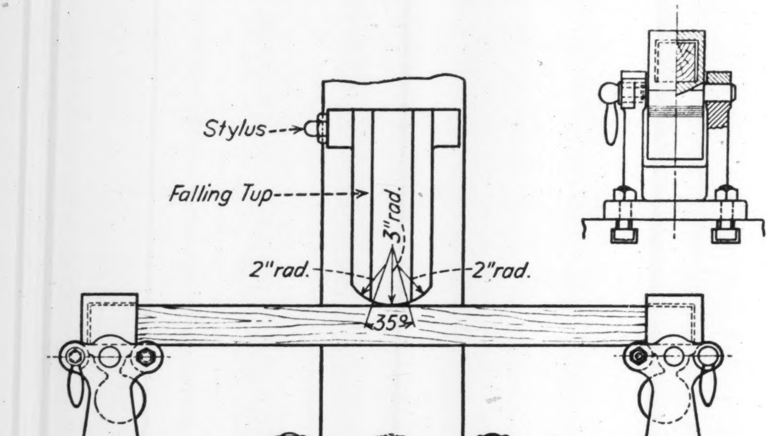

FIG. 3.—Impact Test—Method (*a*).

Wood:

L. J. Markwardt, Senior Engineer, Division of Timber Mechanics, Forest

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[2] 1936 Book of A.S.T.M. Standards, Part II, p. 465.

Products Laboratory, U. S. Department of Agriculture, Madison, Wis., writes as follows:

"The other type of test we have been using to get a measure of shock resistance is the so-called toughness test made with the Forest Products Laboratory toughness machine. ..... It is a single-blow test but differs from the usual impact test in that the load is applied through a cable acting over a drum rather than by direct contact of the pendulum with the specimen. This toughness test is used not only as a means of comparing the shock-resisting properties of different species of wood but also as an acceptance test in the selection of wood for important high-class uses that warrant the expense of such a procedure.

"There are a considerable number of these F.P.L. toughness testing machines in use and some have been constructed in foreign countries. Studies which have been made show an excellent correlation between the results obtained with the Forest Products Laboratory toughness test and the results obtained with the Hatt-Turner impact testing machine."

The above test was described by Wilson in the A.S.T.M. Symposium on Impact Testing of Materials.[3] The design of the machine is shown in Fig. 2.

It will be noted that this test is very similar to a Charpy impact test. It is carried out on unnotched specimens $\frac{5}{8}$ by $\frac{5}{8}$ by 10 in. with an 8-in. span or $\frac{3}{4}$ by $\frac{3}{4}$ by 12 in. with a 10-in. span.

British Engineering Standards Association Methods of Testing Small Clear Specimens of Timber, Specification No. 373-1929, call for impact tests somewhat different from the one described above. Method (*a*) is shown in Fig. 3. The specimen is 2 by 2 by 30 in. and is unnotched. The test span is 28 in. The tup weighs either 50, 100 or 200 lb. The test is described as follows:

"The entire apparatus shall be securely bolted to a solid foundation. The test piece shall be placed in the machine so that the heart side is uppermost. The load shall be applied at the centre of the test piece by the impact of a weight (tup) falling freely from successive heights, increasing by regular increments. The first drop shall be from a height of 2 in. and this shall be followed by drops from heights increasing by 1 in. at a time until a drop of 10 in. has been reached. The height of each successive drop shall then be increased by 2 in., and the test shall be continued until either complete failure or a deflection of 6 in. has been obtained. The maximum drop of the tup shall be recorded."

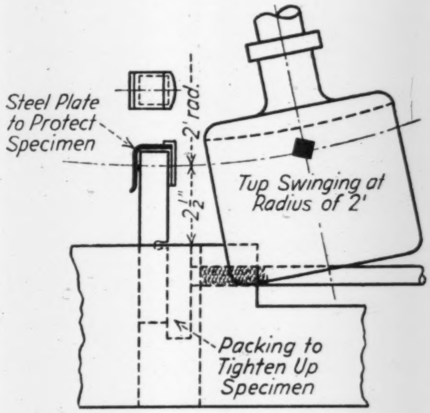
Steel Plate to Protect Specimen
2' rad.
2½"
Tup Swinging at Radius of 2'
Packing to Tighten Up Specimen

FIG. 4.—Impact Test—Method (*b*).

Method (*b*) is shown in Fig. 4. The specimen is of the keyhole-notch type $\frac{7}{8}$ by $\frac{7}{8}$ by 6 in. The notch is a saw cut into a $\frac{1}{8}$-in. diameter drilled hole spaced so that $\frac{1}{2}$ in. of wood remains to be fractured. The specification states: "The machine should be firmly bolted down to a solid foundation. A suitable striking energy for the swinging tup is 20 to 40 ft-lb."

Concrete:

E. F. Kelley, Chief, Division of Tests, Bureau of Public Roads, U. S. Department of Agriculture, Washington, D. C., writes: "In reply, I am sending you enclosed a copy of the December 1937 issue of *Public Roads*, which contains a complete description of our apparatus and the methods of test." The following is quoted from that publication:

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[3] Thomas R. C. Wilson, "Impact Tests of Wood," *Proceedings*, Am. Soc. Testing Mats., Vol. 22, Part II, p. 55 (1922).

"Since the object of the study was to determine the relative effects of sustained and impact forces on the flexural behavior of concrete, the selection of a method of test and the design of the testing machine were guided by the following basic requirements:

"1. It should be possible to apply both sustained and impact forces of known magnitudes to a specimen in such a manner that the only variable present would be the duration of the applied force.

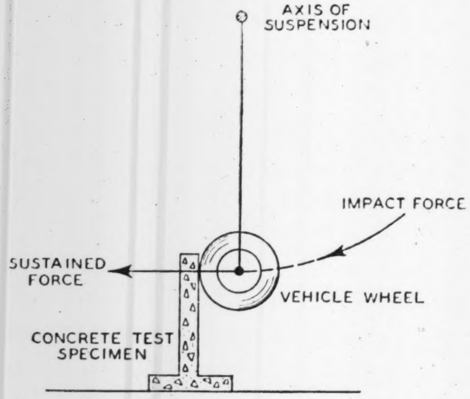

FIG. 5.—Schematic Diagram Showing the Principles of the Test.

"2. The specimens should be flexure specimens of portland-cement concrete.

"3. The magnitude of the forces, the duration of the forces, the masses involved in the impact, the flexing of the concrete and other conditions surrounding the test, should not depart too widely from conditions that obtain when a pavement slab sustains a wheel load.

"4. The testing machine must be of a practicable size.

"5. The force system should be determinate.

"A consideration of these requirements led to the adoption of the method of test shown schematically in Fig. 1 [see accompanying Fig. 5]. A compound pendulum, carrying a vehicle wheel at its center of percussion, is used to apply horizontal forces to a test specimen that is arranged as a vertical cantilever. Impact forces are developed by swinging the pendulum, and sustained forces are developed by exerting a pull on the axle of the wheel on the pendulum.

"The first group of specimens fabricated contained 26 units, the majority of which were of intermediate thickness. Of this group a considerable number have been tested and many thousands of load applications have been made. This experience has demonstrated that the testing equipment is satisfactorily producing the results for which it was designed."

Here we see an adaptation of the impact test with the addition of a static load and with the test not carried to the point of complete failure of the specimen.

Stone:

British Standards Institution Specification No. 595-1935 for Rolled Asphalt calls for an impact test on stone. The specimens are cylinders 1 in. in diameter and 1 in. long. The apparatus is described as follows:

"Any form of impact machine which will comply with the following essentials may be used in making the test:

"(i) A cast iron anvil, weighing not less than 50 kg. (110 lb.) firmly fixed upon a solid foundation.

"(ii) A hammer weighing 2 kg. (4.41 lb.) arranged so as to fall freely between suitable guides.

"(iii) A plunger made of hardened steel and weighing 1 kg. (2.20 lb.) arranged to slide freely in a vertical direction in a sleeve, the lower end of the plunger being spherical in shape with a radius of 1 cm. (0.39 in.).

"(iv) Means for raising the hammer and for dropping it upon the plunger from any specified height from 1 cm. (0.39 in.) to not less than 75 cm. and means for determining the height of the fall to approximately 1 mm.

"(v) Means for holding the cylindrical test specimen securely on the anvil, without rigid lateral support, and under the plunger in such a way that the center of its upper surface shall, throughout the test, be tangential to the spherical end of the plunger at its lowest point."

Enamel:

Navy Department Specifications 52P17 of November 1, 1932, on Primer and Enamel, Bituminous, call for a test for brittleness of the primer and enamel coat as applied in a thickness of $\frac{1}{8}$ to $\frac{3}{16}$ in. to a soft steel plate 12 by 12 by $\frac{1}{16}$ in. as follows:

"The plate shall be supported by a block on the floor so as to give support beneath the point of impact of the falling ball. A 750-g. (1.65-lb.) steel ball shall be dropped from a height of 8 ft. on the coating at a point at least 4 in. from the edge of the plate. Evidences of shattering of the coating from the plate, or radial cracks from the center of the point of impact, shall be looked for. The plate shall then be laid, coating downward, on a board through which a hole about $3\frac{1}{2}$ in. in diameter has been cut. The same steel ball shall be dropped from a height of 8 ft. and shall strike the steel plate over the center of the hole in the board supporting the plate. Any evidences of shattering of coating or the development of cracks shall be looked for."

Automobiles:

H. R. Wolf, Assistant Head, General Chemistry Dept., Research Laboratories Division, General Motors Corp., Detroit, Mich., writes:

"All of our car divisions do a considerable amount of road testing on new models at the General Motors Proving Ground. All of the durability testing includes a certain amount of driving over Belgium block pavement. While this may not ordinarily be considered as impact testing, it does subject the car parts to the same type of impact that occurs in service with the severity and frequency of impact accelerated to a sufficient degree to locate failures in a reasonable length of time without accelerating the test to such an extent as to give test results that are no longer representative of actual service."

Tire Chains:

L. W. Hopkins, Materials Engineer, American Chain and Cable Co., Inc., Bridgeport, Conn., writes:

". . . . substantial changes are now being made in the procedure [for the impact testing of tire chains], and at the present time it would not be in order to give a detailed description until such time as a new procedure had been proven.

"It has apparently been determined that the impact test as a criterion of probable service life of tire chains was satisfactory so long as all factors of material, treatment and design were maintained reasonably constant. The correlation disappears when any of these factors is materially changed."

Small Rollers:

Chrysler Corporation Specifications M.S. 997 on Cageless Bearing and Free Wheeling Rollers cover the analysis of the steel to be used, the hardness of the heat-treated roller and the following impact test:

"*Hammer Test:* The rollers shall be given a hammer test as follows:

"The hammer weight and travel shall be varied according to roller size as listed in the table below. The requirement necessary to pass this test is the number of blows required to shatter or break the roller when in a horizontal position on the anvil."

ROLLER DIAMETER, IN.	HAMMER WEIGHT, LB.	HAMMER TRAVEL, FT.	MINIMUM NUMBER OF BLOWS
0.0625	5	2	2
0.125	10	2	2
0.215	20	2	2
0.327	20	6	2

F. E. McCleary, Metallurgical Engineer, Dodge Brothers Corp., Detroit, Mich., writes:

“This is simply a small drop hammer, as you will see from the weight of the tup, with a single rod vertical bracket carrier from which the tup is released at the specific height. The rollers are laid horizontally on the anvil, one at a time, and subjected to the hammer blows, and, while the specification calls for a minimum number of two blows, at the same time we do not care to have it reach more than four blows, because this indicates, of course, too soft a roller and is again detected by the Rockwell hardness test. In other words, the purpose of the drop hammer was to insure stability against shock, and also be of sufficient hardness to withstand pressure.”

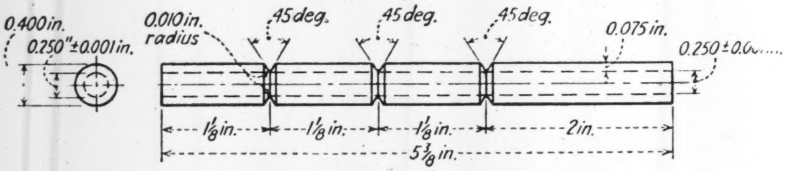

FIG. 6.—Modified Round Izod Impact Test Specimen Used to Determine the Toughness of Stainless Steel.

For machining this bar, a general dimensional tolerance of 0.010 in. is permissible with a tolerance of 0.001 in. on 0.25 in.; there is a fine finish over all and small centers on the ends are permissible.

Spring Stock:

F. E. McCleary of Dodge Brothers writes as follows:

“I might mention in connection with so-called “Utility Testing,” that we have another test we make use of to a large extent for round bar stock, particularly bar stock adaptable to coil spring suspensions, which runs about 0.630 in. in the finished state. We have not been entirely sold on the Izod or Charpy tests, and we made an adaptation of the Izod hammer, rebuilding the vise to accommodate the above-mentioned size test piece, and using a test piece 4 in. long. In the center of the piece we grind a radius groove $\frac{1}{16}$ in. wide, deep and in diameter. Two inches of the test piece is held in the vise, with the groove to the top of the vise, and the blow from the hammer is 1 in. above the groove. Our results have been so consistent, that we use this test to a considerable extent for our own information.”

This same principle for the testing of spring stock was described by the author in an article on “Springs of Stainless Steel” which appeared in *The Iron Age*, October 15, 1936. In that case the springs were being made from round bar stock from which a standard impact test specimen could not be machined. The size and shape of the test specimen adopted is shown in Fig. 6. By means of this very economical test specimen and the use of an Izod testing machine with a suitable change in the vise jaws, it was possible to develop and control a suitable steel and heat-treatment procedure.

Gear Teeth:

B. H. DeLong, Metallurgist, The Carpenter Steel Co., Reading, Pa., writes as follows:

“In the early days of automobile manufacture (1908–1913), the writer’s company supplied many tons of chromium-nickel steel having the analysis of S.A.E. No. 3250 for the manufacture of transmission gears. This material was melted in a crucible furnace, but cast through a ladle practice in heat lots of approximately 3500 lb. each.

Breakage of teeth upon the gears was a considerable problem.

"While it was recognized that, in comparison with present-day standards, there was much lacking from the standpoint of transmission design, gear design, accuracy of heat treatment and other factors, detail study of the problem developed the fact that gears manufactured from certain heats of steel were unusually subject to breakage. Efforts to identify the characteristics of these particular heats by the use of tension tests, slow bending tests, and other procedures which were standard at that time were unsuccessful; tension tests made upon heats which produced good gears and bad gears showed the same results as regards elastic limit, tensile strength, elongation, reduction of area, sclerescope and Brinell hardness.

"A method of test was finally developed which involved the manufacture of a test gear approximately 4½ in. in diameter by 1-in. face. This gear was placed solidly in a cradle beneath two upright supports. A tup was then suspended on rollers between the two supports as a guide, and was dropped from varying heights upon the head of a flat pin, the other end of which was placed in contact with the pitch line of one of the teeth of the gear. The height from which the tup was dropped was gradually increased on successive blows until the tooth was fractured from the body of the gear. A record was then made of the height from which the tup was dropped at the time fracture took place, and this multiplied by the weight of the tup in pounds expressed as foot-pounds.

"It will be observed that this method roughly approximates the clash encountered in the old type of sliding gear transmission when the gears were improperly shifted. This crude method of impact testing produced the results desired. Under this type of test, it was found that gears manufactured from heats of steel which gave poor service would break under a load of approximately 40 ft-lb.; those gears manufactured from heats of steel which gave freedom from breakage in service would withstand from 70 to 90 ft-lb. before rupture took place."

Cap Screws:

Alvan L. Davis, Research Engineer, Scovill Manufacturing Co., Waterbury, Conn., writes as follows:

"A simple way of applying a shock, or blow, to the under side of the head of a cap screw, is shown by the accompanying sketch [see Fig. 7]. The operation is as follows:

"A sturdy washer (marked *A* in the sketch) is first slipped on the bolt, or cap screw, which is then inserted in place, on the under side of the seat of a "stool," (designated by *B* in the sketch). A nut *C* is then screwed to hold the bolt securely in place.

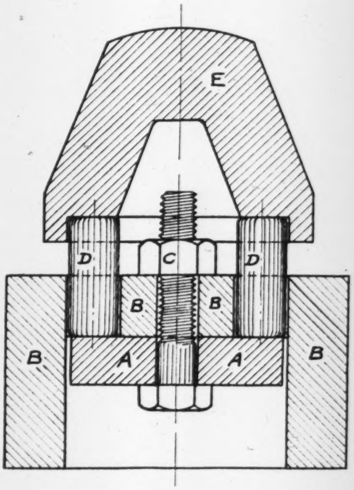

FIG. 7.—Method of Applying a Blow to the Under Side of the Head of a Cap Screw.

"There is a central hole in seat *B* for the bolt to pass up through. Also there are three other symmetrically disposed holes in the seat of the stool, just inside the peripheral shell which forms the 'legs' of the stool. Through these three holes slide three hardened steel thrust pins, *D*, with easy clearance.

"A cap *E* rests upon the three pins, and is held centered upon them by a slight flange. The top of the cap has a central boss to receive the blow of a hammer, or tup, which is allowed to drop from a height that will give enough velocity to make the test "snappy." The tup is of adequate weight to provide energy sufficient to break the cap screw being tested by a single blow.

"The location, and kind of fracture, determine the character of the cap screw. For example, a properly made cap screw always breaks through the threaded portion of the stem, and with an oblique shear fracture which intersects several convolutions of the thread, rather than squarely across at the bottom of a single thread. The head never parts from the screw at the shoulder.

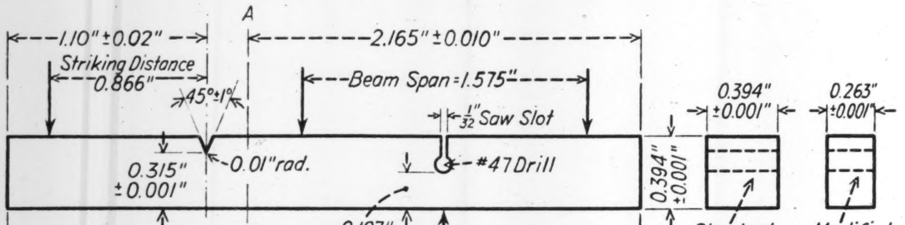

FIG. 8.—Combination Izod-Charpy Impact Specimen.

NOTE.—The combination Izod-Charpy specimen is first prepared with only the Izod V-notch. After making the Izod test, the fractured end is cut off along the line A—A' and the remainder notched for the Charpy test in the usual manner.

"In a screw made of wrong material, or with improperly upset head, the head will fly off at the shoulder, and there is no elongation observable at the plane of fracture.

"In practice the above device is used under a small stamp, or drop hammer. With two operators 10 screws can be tested in about 10 min. The cost of the device is about $30, as but few surfaces require accurate finish. It has proved satisfactory over a period of two years in use."

Pipe and Tubing:

Various types of expanding mandrel and hammering tests are used on pipe and tubing of different kinds. For sizes up to about 6 in. in diameter, extra heavy, the wall thickness of pipe and tubing is insufficient to yield any of the present-day standard impact test specimens. Nevertheless, the presence or absence of undue brittleness is often of vital importance. This can often be tested for by using "miniature" specimens machined from the walls and tested in a manner similar to that used on a full-size specimen in an Izod or Charpy type of machine.

Tests of this type with a modified specimen are used by one of the large tube companies and have apparently proven their value. The location, size and shape of the specimen is shown in Fig. 8 supplied by the National Tube Company Research Laboratory.

Thin-Wall Tubing:

Thin-wall tubing such as used in airplane construction and for steel golf-club shafts does not permit of the use of a "miniature" specimen machined from the wall stock. Here an entire tube section, however, can be given an impact test. Either the Charpy or the Izod type of test may be used if the tube section is uniform throughout the length of the specimen. Where the tube section is not uniform, as in tapered golf shafts, the Izod type of test proves the most adaptable. Any of the Izod type of testing machines may be used.

Special vise jaws should be provided to hold the specimen rigidly in position. The test, as applied to golf-club shafts, has proven very useful in controlling both the quality of the material and the efficiency of the heat treatment applied.

Rails:

British Standards Institution Specification No. 11-1936 for Flat-Bottom Railway Rails include a pendulum impact test. The arrangement for test is shown in Fig. 9. Test pieces are required to withstand an appropriate blow without fracture. The blows specified range from 14 ft-lb. to 83 ft-lb. according to the size of the rail. The test is described as follows:

> "If the engineer (or purchaser) requires pendulum impact tests, they shall be carried out in approved impact testing machine of the pendulum type. The testing machine shall be so designed that when a test piece, as described below, is suitably clamped by the flange to the base of the machine, the point of impact shall be on the side of the head at its lower radius.
>
> "Samples for testing shall be prepared in the following manner. A short length shall be cut off at the hot saw from the tail end of the last rail of a selected ingot to pass through the rolls or, with the consent of the manufacturer, from that part of the crop immediately adjacent, and shall be stamped hot near one end with the cast number. A pair of adjacent test pieces each 1 in. (25.4 mm.) thick shall be cut cold from the middle portion of this short length, but before cutting, the cast number shall be transferred to the side of the head of each test piece. All the stamp marking specified above shall be carried out on the same side of the rail as the branding. A tolerance not exceeding plus or minus $\frac{1}{32}$ in. (0.79 mm.) shall be allowed in the thickness of each test piece.
>
> "One test piece of a pair so cut shall be clamped in the testing machine so as to receive the blow from the falling weight on the side of the head bearing the stamp markings; the other test piece of the pair shall be tested in the same manner, except that the blow shall be applied to the unstamped side of the head."

IMPACT TESTING PROBLEMS

Each example selected for description or discussion in this paper represents the result of considerable study applied to a specific problem. Many more examples of how specific problems have been answered could have been included. Many problems still await solution.

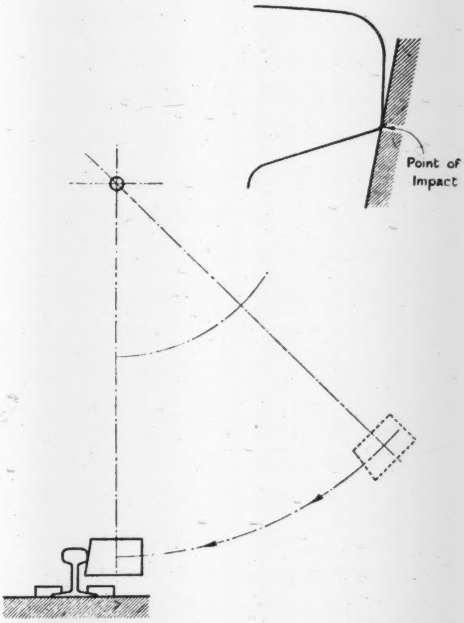

FIG. 9.—Pendulum Impact Test.

Warren E. Emley, Chief, Organic and Fibrous Materials Division, National Bureau of Standards, Washington, D. C., writes as follows:

> "In our work on the development of a substitute for glass for windshields for airplanes, impact resistance is very important. As the problem was put up to us originally, it was to find a material which would withstand the impact which occurs when an airplane traveling 200 m.p.h. meets head-on with a 4-lb. duck traveling at 60 m.p.h.

From all of our theoretical considerations we have been unable to find any small convenient test method which would give results which could be interpreted in terms of actual service. Since tests simulating actual service conditions are laborious and expensive, we would like very much to find some simpler test which would give reliable and comparable results. While the matter may sound amusing, it is every year the cause of a few fatal accidents.

"In the testing of molded plastic articles for acceptance under contract, the ability to resist impact is frequently considered important. I recently visited a laboratory doing a great deal of routine testing of this nature and found that they were making four impact tests on every material submitted, the impact bars being specially molded at the time articles purchased were being molded. The four tests were the Charpy and Izod both notched and unnotched. From the information gained from the four results, plus consideration of the fact that the specimen tested was not one of the articles called for in the contract, plus 20 yr. experience, the laboratory felt that it could estimate the probable resistance to impact of the articles delivered. You can see that this is a very roundabout and unsatisfactory way of obtaining information."

CONCLUSION

An attempt has been made to show the great value and wide adaptability of various forms of non-standard impact tests. It is hoped that this discussion will stimulate further uses of this type of test. In cases where the standard types of impact testing machines on the market are not suitable, special types can be built. The suggestion is made that special adaptations of standard machines should be preferred over entirely new machines. Special vise jaws for oddly shaped or non-standard size specimens can be fitted readily to most of the machines on the market. Modifications in the striking tup or hammer are not difficult to make.

The fact that the numerical results obtained in a non-standard impact test are not comparable to numerical results on standard tests should not act as a deterrent.

Particular emphasis may be laid on the subject of expensive forms of test specimens. A rectangular test bar is often many times as expensive to machine as a cylindrical one. A notch which requires the use of a special milling cutter is much more expensive than a notch which can be turned in a lathe or cut in with a grinding wheel. Results obtainable with an inexpensive specimen shape and notch form on the Izod type of specimen have been shown to be of considerable value in industry. The expensive refinement of a milled notch in one side only and of a flattened striking area on the specimen have been shown to be unnecessary.

A plea is made that those engaged in the field of "scientific" impact testing take a broad view of the entire "art," promote the "art" as a whole, refrain from attempts to standardize on too limited a number and expensive forms of test specimens, and consider the utility or non-standard impact test of today as the possible standard of tomorrow.

DISCUSSION

Mr. L. B. Tuckerman.[1]—This is a very welcome addition to our knowledge of non-standard impact tests. Practically everyone who has been engaged in materials or structural tests for any length of time has been faced with the necessity of devising such tests. These examples of how such problems have been solved will make it easier for others to solve similar problems.

Usually when such tests are made the man who makes them has or thinks he has a fairly clear idea of their relation to the particular use of the material or structure in which he is interested. If those of us who stand on the side lines are to evaluate these tests we should also know not merely how the tests were made but what were the correlations with service records which showed their value. It would help us much in understanding the significance of impact tests.

In one example this correlation is discussed and the discussion shows how illuminating such information can be. The statement is: "It has apparently been determined that the impact test as a criterion of probable service life of tire chains was satisfactory so long as all factors of material, treatment and design were maintained reasonably constant. The correlation disappears when any of these factors is materially changed."

These particular impact tests, at least, apparently have much in common with indentation tests. By themselves they afford no reliable comparison between the serviceability of materials differing much in composition or type of treatment. It seems to me probable that the chief value of impact tests, as it is of indentation tests, will always be in the comparison of supposedly similar pieces of similar material, similarly treated, when the comparison has been checked with service records.

One statement in the paper should not pass without comment. "The nature of the notch or notch effect is usually determined by *unavoidable* [italics mine] circumstances or details of the design." At the National Bureau of Standards we have just finished investigating two serious failures, one of which cost several human lives. In these cases the notch was *not* unavoidable.

Any engineer familiar with fatigue testing and the danger that lies in sharp reentrant angles, rough machined fillets and thin outstanding fins should have known that in these cases such notches *must* be avoided if the structure was to be safe.

No one today is justified in using the words "*unavoidable notch*" in an important structure subject to vibration or impact. Too many people have already been killed by "*unavoidable notches*."

Mr. H. F. Moore.[2]—I wish to add my word of congratulation to Mr. Tour. He has contributed further items to the available lists of significant uses of the impact test. Even if the full scientific implications of the impact test are not understood, it seems reasonable that a material which will withstand more energy before fracture than another should have that fact noted as a point in its favor. Mr. Tour's careful distinction between carefully standardized, scientific impact tests and simpler service tests is worthy of being read three times and then thought over.

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[1] Assistant Chief, Division of Mechanics and Sound, National Bureau of Standards, Washington, D. C.

[2] Professor of Engineering Materials, University of Illinois, Urbana, Ill.

I wish to suggest a test which can sometimes be used in place of, or still better as supplementary to, the impact test—the static test in which a complete load-deformation diagram to fracture is obtained, and the energy for fracture measured by the areas under the load-deformation diagram. In some cases it is possible to make such a static test when impact apparatus is not available, and unless the much discussed "critical speed" is exceeded, the *energy* as measured in the static test will indicate the same property as the energy measured by the impact test. A case in point is a static bend test for steel rails in place of, or supplementary to, the present standard drop test.

If the critical speed is not exceeded, usually it is found that more energy is required for fracture in an impact test than in a static test, because of the greater "leakage" of energy to machine parts of the impact test. The impact machine developed by Professor Southwell of Oxford University is of interest in this particular. If, however, an impact test showed markedly *less* energy for fracture than a static test it would seem highly probable that the material had a critical speed below that used in the particular impact test used.

MR. SAM TOUR.[3]—I wish to thank the speakers for their kind remarks. I have no discussion to offer on Mr. Moore's suggestion about using the area under the load deformation diagram of a static test carried to the fracture point, as indicative of impact strength. Possibly it can be used. In regard to Mr. Tuckerman's remarks, I wish to agree with them to a large extent; when you have widely different materials and widely different treatments, the results are not comparable. In regard to tire chains, I believe that these are a specific example of where, with changes in material and heat treatment, numercal results are no longer of value for comparison. I also wish to agree with him in some of his statements about some of these avoidable notches in the work of engineers and architects. While I often find that in machine and structural design certain notch effects must be present that cannot be avoided, it is also very true that we have them where they could have been avoided. We cannot stress too much the need for engineers and architects to eliminate reentrant angles and to eliminate rough machining in angles and to use good, generous fillets.

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[3] Vice-President, Lucius Pitkin, Inc., New York City.

THE IMPACT TESTING OF PLASTICS

By Robert Burns[1] and Walter W. Werring[1]

Synopsis

There are approximately 8,000,000 molded phenol plastic telephones in use throughout the Bell System. In the very nature of things many of these instruments, during their service life, will be subjected to impact forces resulting from accidental dropping. The extent to which actual breakage occurs, and consequent loss of service to the subscriber and expense involved in making the necessary replacements, is determined largely by the impact strength of the molding material. This large interest in the impact strength of plastic materials led to the development by the Bell Telephone Laboratories of pendulum type impact machines of suitably low capacity for tests of molded specimens of plastic materials, and of other equipment for laboratory impact tests of assembled apparatus. Studies and control of the impact properties of these materials have been carried on for a number of years and improvements indicated by laboratory tests have been substantiated by accurately kept field experience records.

This paper presents the results of this experience. It also reviews in part the work of the Society's Committee D-9 and attempts to point the way toward further progress in the art of impact testing of plastics. The important effect of moisture on the impact strength of molded telephone apparatus is shown, and it is demonstrated that reproducible results on test bars of phenol plastic can be obtained only when this feature is carefully controlled. For some of the thermoplastics, control of temperature as well as moisture is necessary in precision testing.

The excellence of the Izod or Charpy test using notched bars is reaffirmed but it is also shown that no single method can be expected to suffice for all the plastics. A group of three tests is suggested, and evidence is presented to show that a material proven to be strong by these tests, when properly molded, will invariably produce a strong product. These data also make manifest that a properly molded product, that is, one that truly develops the strength properties of the raw material, is the *sine qua non* of plastics testing.

Tests to determine the resistance of organic plastics to impact have been largely a development of the last 15 yr. During this time the Society's Committee D-9 on Electrical Insulating Materials has actively promoted the investigation of impact testing of all types of plastic materials. The earliest work was done with increment drop tests because sufficiently sensitive pendulum machines were not available. However, the desire to determine the actual energy absorbed in breaking the specimen led to the development of pendulum type machines of suitably low capacity.[2] The need for special machines for plastics arises from the fact that the most commonly used materials have impact values between 0.10 and 1.00 ft-lb. for a specimen $\frac{1}{2}$ in. square in cross-section notched to a depth of 0.100 in. A number of other investigators built machines patterned after these early ones and the development of methods of test to insure reliable and reproducible results was begun. The standard methods are now embodied in A.S.T.M. Tentative Methods D 256 - 34 T.[3]

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[1] Members of the Technical Staff, Bell Telephone Laboratories Inc., New York City.

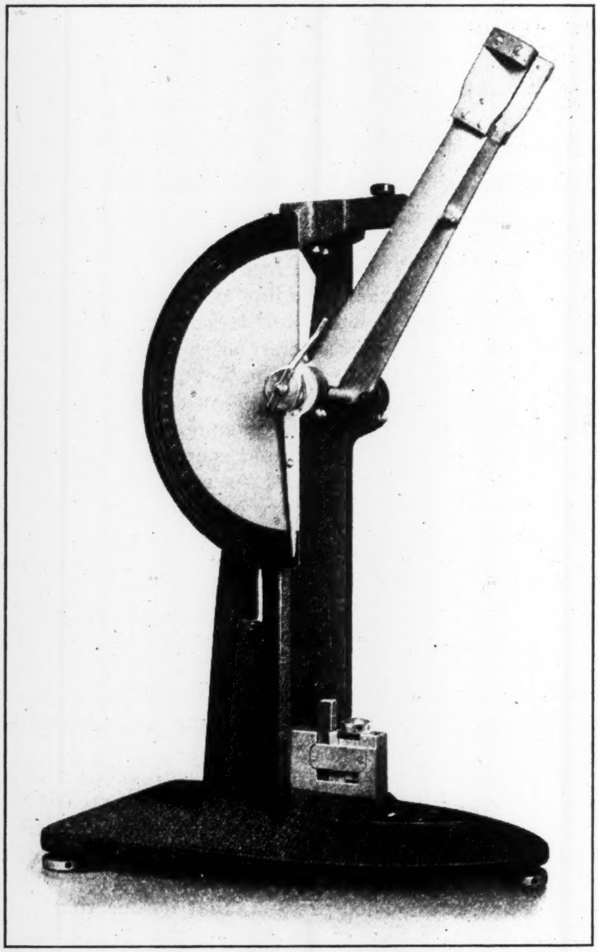

FIG. 1.—2-ft-lb. Izod Impact Machine.

Test Machine:

The earliest results indicated the existence of a velocity effect and it was necessary to establish a standard velocity of impact. The possible height of fall in machines of the small capacity required is limited by mechanical difficulties of design. Opposed to this it was generally considered desirable to use a high velocity of blow to reveal fully the brittleness of the material. As the height of fall of the pendulum in the usual Izod test is 2 ft., this was adopted as a practical compromise for tests of both Izod and Charpy types.

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[2] W., W. Werring, "Impact Testing of Insulating Materials," *Proceedings*, Am. Soc. Testing Mats., Vol. 26, Part II, p. 634 (1926).

[3] Tentative Methods of Testing Electrical Insulating Materials for Resistance to Impact (D 256 - 34 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 995 (1934); also 1937 Book of A.S.T.M. Tentative Standards, p. 1023.

Changes in the capacity of the machines between 2 and 5 ft-lb. were not found to affect the results appreciably, but a machine of only ½ ft-lb. capacity indicated absorbed energy values as much as 25 per cent lower than those of the 2 ft-lb. machine. This was apparently a further manifestation of the velocity effect resulting from the deceleration of the light-weight hammer during fracture. Increasing the weight of the hammer of the smaller machine without other changes in design eliminated this difference.

As a result of these experiments, the design of the usual machine became fixed at 2 ft-lb. capacity at a height of fall of 2 ft. although increases to 4 or 5 ft-lb. may be made by adding supplemental weights to the sides of the hammer. An improved design, Fig. 1 (Izod form), embodying the experience of a number of years with the old type[2] was developed and is now in use at several laboratories.

Test Specimens:

Molded plastics present a difficult problem in the production of truly representative test specimens. Differences in the method of molding the specimens may produce variations in properties which will not be representative of a properly molded useful product and conversely variations in the regular manufacturing process may result in product differences not inherent in the material. It is therefore our practice to specify in detail the mold and molding process to be used for the production of specimens for impact tests. By these means reproducible test results can be obtained consistently and it is only by such control that the test results will be indicative of the useful properties of the material. The problem of insuring a uniformly good product from uniformly good raw material would require the same precision of control of manufacture as that used in producing the test specimen and it is manifestly a more exacting job. This difference between specimen and product makes it necessary in the case of important molded apparatus to supplement raw material tests with impact tests of the finished product.

The choice of specimen dimensions involves the characteristics of the materials in a way not usual in metals. In many plastic materials, the maximum thickness that can be properly molded is definitely limited and such materials are used within this limitation. Molded test specimens should therefore be of similar thickness to be representative of the material. However, in testing materials for use in a particular piece of apparatus in which the thickness of section is fixed by the design, the specimen used must then conform to the thickness actually used even though this may be a size which will not develop the optimum impact properties of the material. This latter is the case for much important telephone apparatus so the specimen used in our tests is in practically all cases ½ by ½ in. in cross-section. This is the specimen originally standardized but present A.S.T.M. practice[3] also permits use of specimens ½ in. by any desired smaller dimension. The thin specimens are struck on the narrow side.

Efforts to relate the cross-sectional dimensions of specimens, either notched or unnotched, to the energy expended in breaking them have been generally unsuccessful. For some materials the energy required to break notched specimens varies roughly in proportion to the breadth of the specimen multiplied by the square of the depth back of the notch, but this relation does not hold closely enough to permit accurate correction of even small differences in specimen size. It is therefore permissible only to state absorbed energy in terms of a standard specimen and it is necessary to hold the specimen size to fairly close limits. For the notched test the over-all dimensions of the specimen may vary considerably, but the thickness back of the notch should be controlled to within ±0.002 in. However to avoid variations in clamping in the Izod test, individual bars should not vary in thickness from end to end more than 0.002 in. For unnotched tests the over-all thickness becomes of greater importance and must be held to a definite maximum for specification purposes.

Type of Notch:

When the velocity of impact is high, the particular type of notch used is not important so long as it is sufficiently deep and sharp. In early tests various notches were tried, but the notch now used for both Izod and Charpy tests is the 45-deg. V notch with 0.010-in. radius at the bottom. This is the same shape as the standard Izod notch for metals, but a depth of 0.100 in. is used instead of 0.079 in. because of the larger cross-section of the plastic specimen.

The effect of the sharpness of the bottom of a 0.100-in. deep notch on the absorbed energy value of a phenol plastic is shown in the following table:

RADIUS AT BOTTOM OF 45-DEG., 0.100-IN. DEEP, V NOTCH, IN.	IZOD TEST ABSORBED ENERGY, FT-LB.
0.010	0.135
0.020	0.133
0.030	0.134
0.040	0.141
0.050	0.165

Notch Effects:

Both notched and unnotched specimens are used in impact tests of plastics. Both indicate differences in materials which are not apparent from the usual static tests but which become obvious in the service behavior of the material. In addition the notched-bar test indicates some established differences not always shown by the unnotched test. In the case of molded telephone apparatus, impact forces during service life are usually those resulting from accidental dropping. The exact circumstances are therefore variable and uncertain. Likewise the initial stress condition in the assembled product may vary widely and is practically unpredictable. Consequently it is difficult to establish the relative significance of individual properties for a particular use. Of course, the material that excels in all properties will be the most desirable. While obviously not the exclusively significant test, the notched test is probably the most significant single test of the impact resistance of molded plastics.

A range of widely different types of phenol plastics varying from the ordinary grade of material to the least brittle material of the type is represented by the following data. In these materials the flexure strength gives no indication of impact resistance as measured on test bars.

MATERIAL	NOTCHED IZOD, ENERGY, FT-LB.	FLEXURE TEST, MODULUS OF RUPTURE, LB. PER SQ. IN.
A.........	0.136	9600
B.........	0.172	8780
C.........	0.248	5180
D.........	0.686	8150
E.........	1.65	9930

There is unfortunately no theoretical method of calculating the effect on the strength of a part of these differences in the energy absorbed in breaking specimens. However, some idea of the magnitude of the difference which may be significant in plastic materials is indicated by the results of tests of a molded housing. It contained apparatus bringing the total weight to about 7 lb. Made of material with a notched Izod strength of 0.2 ft-lb. it would occasionally crack when repeatedly dropped from a height of 3 ft. to a soft wood floor. When made of material of 0.4 ft-lb. strength it could be dropped practically indefinitely from the same height on a concrete floor.

TABLE I.—STRENGTH RATINGS OF HARD RUBBER AS INDICATED BY IMPACT TESTS AND EXPERIENCE.

Rating	Charpy Plain			Charpy Notched			Izod Notched		
	Material	Impact, ft-lb.	Experience Rating	Material	Impact, ft-lb.	Experience Rating	Material	Impact, ft-lb.	Experience Rating
1	A	>3.0	High	A	0.372	High	A	0.295	High
2	C	2.64	High	B	0.270	High	B	0.287	High
3	D	2.17	High	C	0.252	High	C	0.272	High
4	E	1.86	Medium	D	0.235	High	D	0.234	High
5	F	1.37	Medium	E	0.229	Medium	E	0.230	Medium
6	B	1.35	High	F	0.220	Medium	F	0.217	Medium
7	H	1.32	Fair	G	0.213	Medium	H	0.182	Fair
8	I	1.28	Fair	H	0.195	Fair	G	0.158	Medium
9	G	1.10	Medium	I	0.180	Fair	I	0.135	Fair
10	J	0.70	Low	J	0.154	Low	J	0.109	Low

One comparison of the results by notched and unnotched bar tests is shown in Table I. These data are from tests of a variety of hard rubber compounds which were rated for resistance to impact breakage and the general quality of their mechanical properties by engineers responsible over a period of years for their compounding. It will be seen that the notched-bar Charpy results in every case agree with the previous experience judgment on the material whereas the results on the unnotched Charpy do not. The notched Izod agrees with the notched Charpy except for the minor interchange of ratings 7 and 8.

Types of Tests:

Both Charpy and Izod tests are used and though the actual values of absorbed energy obtained are slightly higher in the case of the Charpy the relative indications are generally similar. On account of the large sample there is some tendency for the broken halves to interfere with the Charpy hammer. The Izod test has the further advantage that it permits two tests per molded bar instead of one and in fact permits test of the broken halves of bars previously tested for flexural strength, which does not affect the impact values and is a considerable economy. It is for these reasons that we use the Izod more generally than the Charpy type machine.

Effect of Moisture:

Studies made some years ago of the factors likely to cause variability in impact test results revealed that the moisture content of the specimen at the time of test greatly affected the results. Differences in moisture content of a few tenths of a per cent which will result from only a few days of high humidity caused differences of as much as 25 to 50 per cent in notched-bar strength. Obviously some "conditioning" before test would be necessary to eliminate the effects of the variable history of the specimens if the data were to be truly representative of the material.

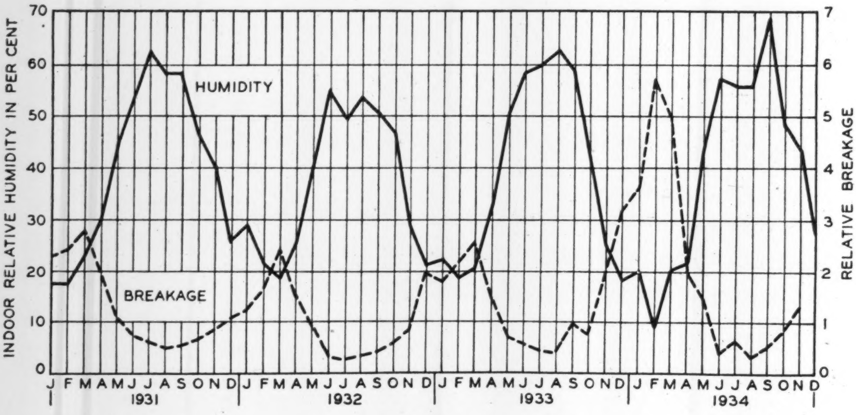

FIG. 2.—Apparatus Breakage and Indoor Relative Humidity, New York City.

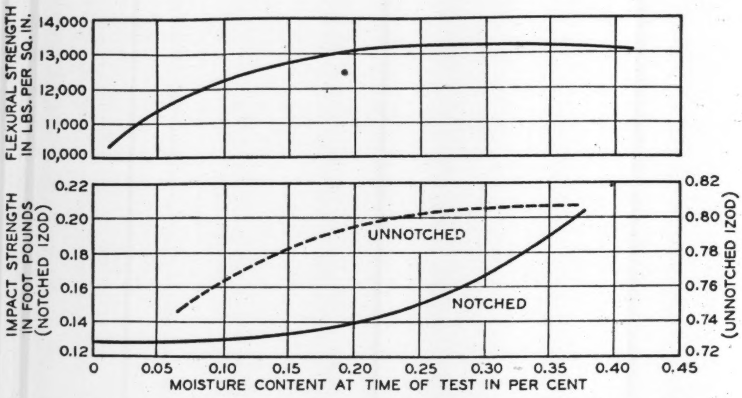

FIG. 3.—Effect of Moisture Content on Physical Properties of Phenol Plastic.

This effect of moisture, first observed in test specimens, has since been correlated with actual breakage of molded parts in service. It has been established as the cause of the seasonal variations in breakage rates observed where molded apparatus is subjected to artificial heating in winter as in New York City and lacking in climates where heating of buildings is not necessary. The relation between breakage rates experienced and the relative humidity of the ambient atmosphere over a period of years is shown in Fig. 2. The effect of moisture upon the properties of the material used in the apparatus covered by Fig. 2 is shown by Fig. 3 and it will be noted that the continuous increase in notched-bar strength is most striking. The adoption of materials indicated to be improvements by the impact test has resulted in considerable reduction in breakage in service with substantial savings. For example in the case of the apparatus represented by Fig. 2 the reduction of relative breakage rate by improvement in impact properties is shown by Table II.

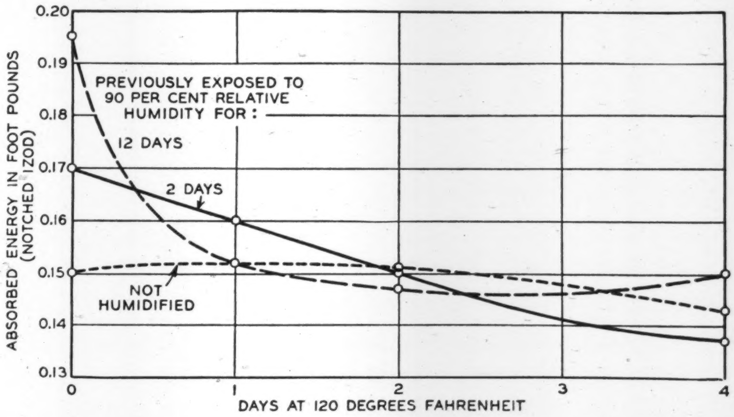

FIG. 4.—Eliminating the Effects of Previous History of Phenol Plastic by Exposure to 120 F.

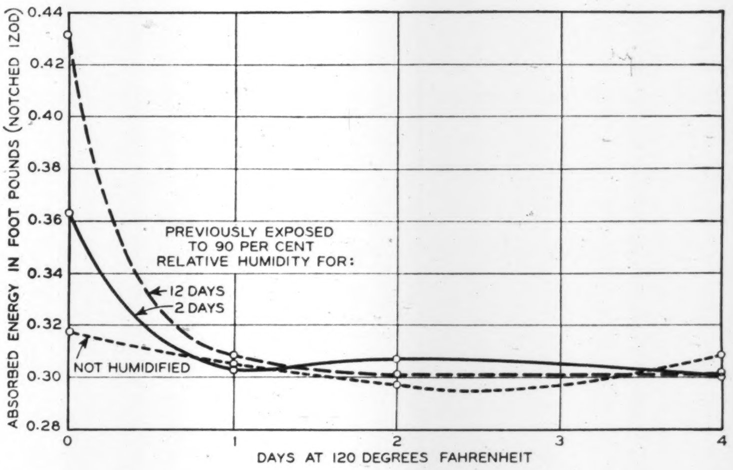

FIG. 5.—Eliminating the Effects of Previous History of Phenol Fiber (Laminated Phenolic) by Exposure to 120 F.

Effect of Conditioning:

Conditioning Before Test.—The marked effect of moisture on the impact strength has been recognized by Committee D-9 and it has been found necessary to condition test bars prior to test to make possible a reasonable degree of reproducibility. Since the user of plastic materials is primarily interested in how the material will perform under adverse service conditions, various drying treatments were investigated and heating at 50 C. (120 F.) for 48 hr. was adopted. This treatment brings the specimen to a state comparable with the dryest it will be in normal indoor service and has been shown, by a comprehensive series of tests made by four laboratories, to erase or nullify the previous atmospheric history of the specimen. Other treatments such as exposure to low humidities at room temperature will also nullify previous history but such procedures are generally considered impracticable due to the long time necessary to produce satisfactory results.

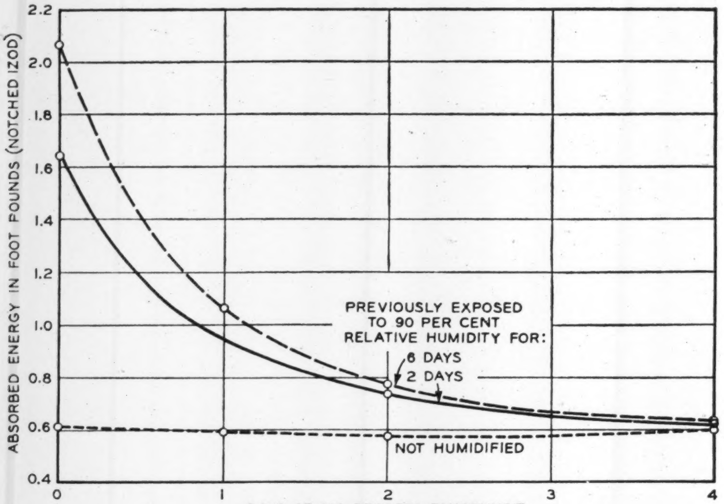

FIG. 6.—Eliminating the Effects of Previous History of Cellulose Acetate by Exposure to 120 F.

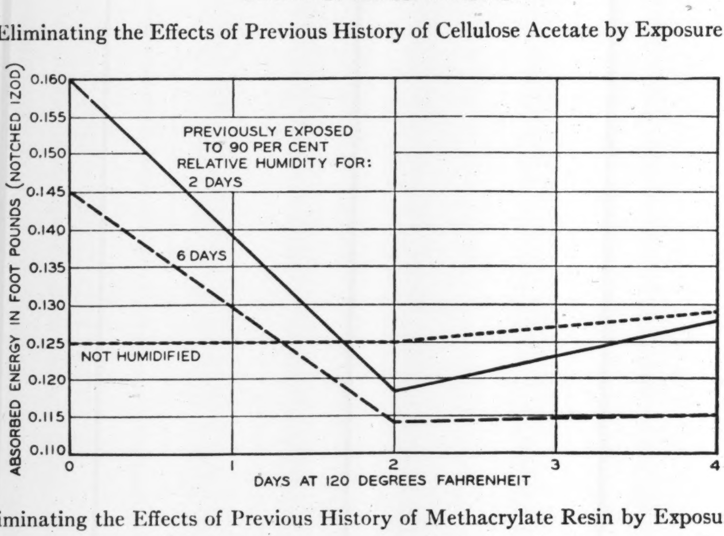

FIG. 7.—Eliminating the Effects of Previous History of Methacrylate Resin by Exposure to 120 F.

The effect of conditioning on the notched Izod impact strength of phenol plastic, laminated phenolics, cellulose acetate and methacrylate resin is shown in Figs. 4, 5, 6 and 7 respectively. These data show the substantial effect of moisture, especially on the cellulose acetate, and also the length of time at 50 C. (120 F.) to bring them to a condition where reproducible values may be obtained. Examinations of the strength values before and after relatively long periods of heating at this temperature reveal no measurable effect on the material other than removing the moisture.

In the above tests the phenol fiber specimens were tested edgewise since

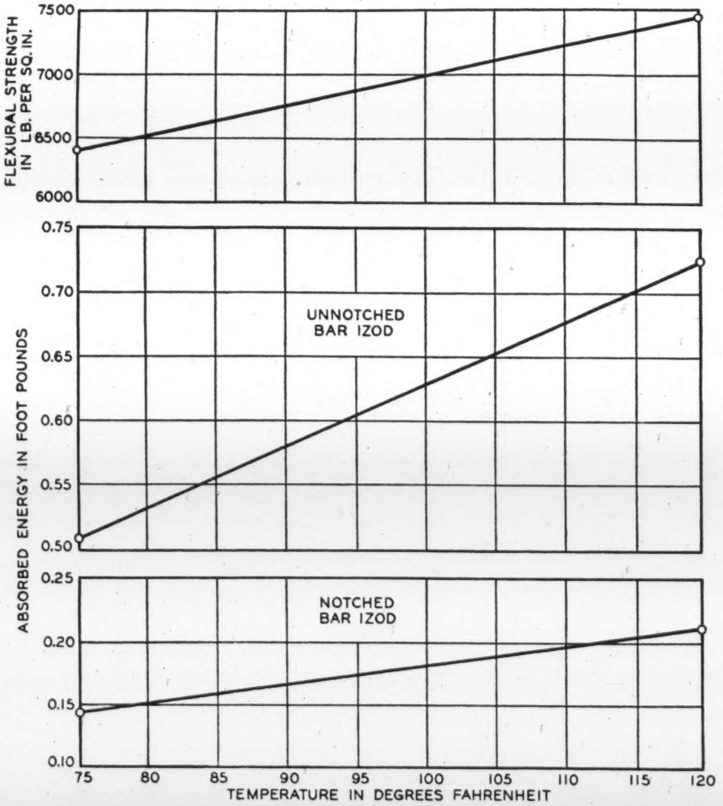

FIG. 8.—Effect of Temperature on the Notched-Bar Izod, Unnotched-Bar Izod, and Flexural Strength of a Commercial Cellulose Acetate.

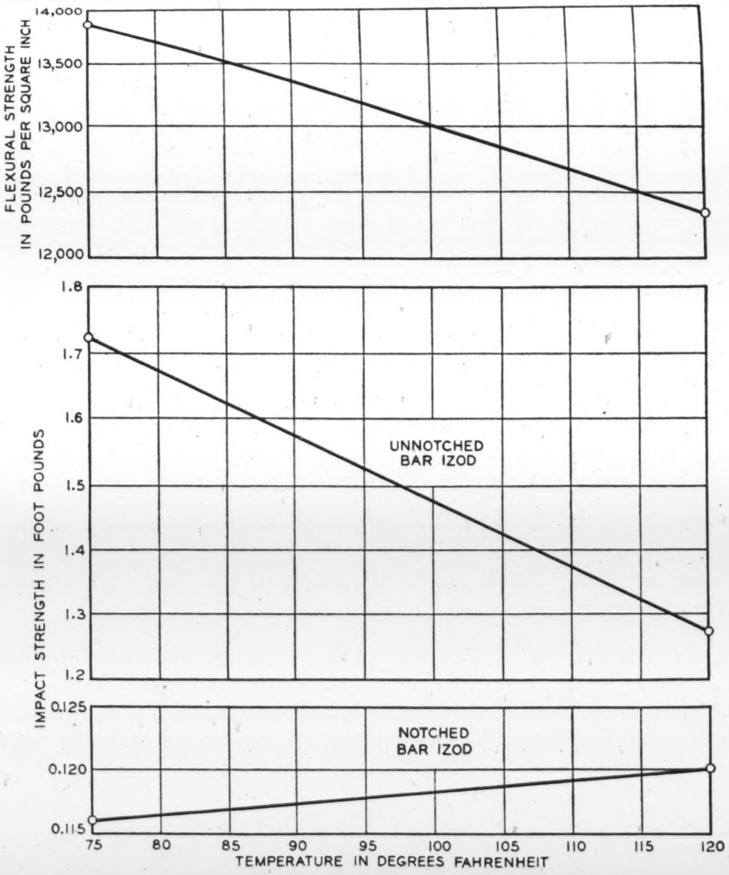

FIG. 9.—Effect of Temperature on the Notched-Bar Izod, Unnotched-Bar Izod, and Flexural Strength of Methacrylate Resin.

this provides more consistent results than can be obtained with the flatwise break and indicates the minimum strength of the material.

Condition at Test.—Since the impact strength of the phenolics is not critical to those temperature variations normally encountered in the usual laboratory it has sufficed to permit testing at any convenient room temperature, the bars being broken immediately upon removal from the desiccator in which they have cooled. The situation is somewhat changed, however, for the thermoplastics which naturally are more responsive to temperature. Figures 8 and 9 show the effect of temperature on the notched Izod, unnotched Izod and flexural strength of a commercial cellulose acetate and a methacrylate resin. Lack of a fixed relationship between flexural strength and impact strengths appears to characterize both these materials.

TABLE II.—RELATION BETWEEN NOTCHED-BAR IZOD, APPARATUS STRENGTH TESTS, AND FIELD EXPERIENCE.

	Notched-Bar Izod Impact Strength on Standard A.S.T.M. Test Bars, ft-lb.	Strength of Telephone Apparatus as Determined by Height of Fall Necessary to Break, in.	Relative Breakage in Field.
FOR DISSIMILAR PHENOLICS			
Standard wood flour filled material	0.140	30	1.00
High flock filled material	0.240	60	0.40
FOR A SINGLE PHENOLIC OF APPROXIMATELY 15 PER CENT FLOCK			
Batch 1	0.150	42.0	
Batch 2	0.153	43.0	
Batch 3	0.157	44.0	
Batch 4	0.159	44.5	
Batch 5	0.164	45.5	

Inadequacy of Single Tests:

In the early days of the plastics industry the phenol plastics were used most generally for applications where physical or electrical properties were of paramount importance. Of the phenol plastics then available the product embodying approximately 50 per cent resin and 50 per cent wood flour was used almost exclusively. Into this relatively simple situation, comprising somewhat uniform resins, fillers and plasticity properties, a single impact test fitted surprisingly well for it was found that both the Charpy and Izod tests on $\frac{1}{2}$ by $\frac{1}{2}$ by 5 in. bars would predict the service strength of molded parts with reasonable accuracy.

TABLE III.—ILLUSTRATING THE LACK OF RELATION BETWEEN THE RESULTS OF ANY SINGLE IMPACT TEST ON STANDARD TEST SPECIMENS OF FOUR PHENOLIC MATERIALS AND RESULTS OF TESTS ON THE COMPLETED APPARATUS FABRICATED OF THE SAME MATERIAL.

Material	Standard A.S.T.M. Notched-Bar Izod, ft-lb.	Unnotched-Bar Izod, (Standard A.S.T.M. Except Unnotched), ft-lb.	Increasing Blow Notched-Bar Izod, in-lb	Increasing Blow Unnotched-Bar Izod, in-lb.	Unnotched-Bar Charpy, ft-lb.	Strength of Telephone Apparatus as Determined by Height of Fall Necessary to Break.
A	0.247	0.829	1.5	6.3	1.145	60
B	0.189	0.735	1.1	6.3	1.035	60
C	0.235	0.584	1.4	5.0	0.925	45
D	0.140	0.619	1.0	6.2	0.971	30

Table II gives data showing the relation between the notched bar Izod test and the strength of telephone apparatus as judged by tests on the completed apparatus and by field experience. Laboratory tests of the completed apparatus are made using the device shown in Fig. 10. This is a metal chute and anvil, and strength is measured by the height of fall at which break occurs in an increment drop test.

It was to be expected that this fortunate situation would not continue indefinitely. The use of mixtures of wood flour and cotton flock for applications requiring high-strength phenolics, the introduction of material of various plasticities to permit a wider assortment of molding techniques, and the increasing use of the thermoplastics for mechanical and electrical as well as decorative purposes, introduced into the plastics picture a group of mechanical properties far too complicated for evaluation by any single test, however well conceived or skillfully manipulated.

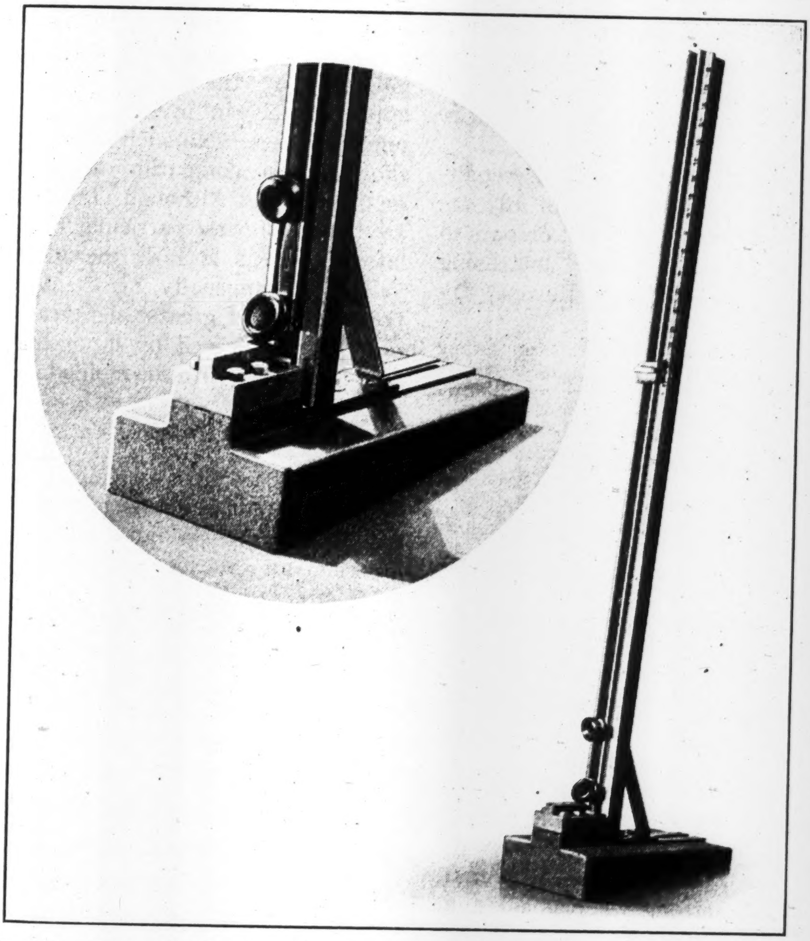

FIG. 10.—Device for Making Laboratory Tests on Completed Telephone Apparatus.

Table III illustrates the lack of relation between the results of any single impact test on standard test specimens of four phenolic materials and results of tests on the completed apparatus fabricated of the same materials. These materials may be described briefly as follows: material A is a moderately high impact grade combining a relatively soft resin with a high cotton filler whereas material B is a stiffer resin with about one-third the flock content of material A. Material C is similar to A except of a somewhat differently processed compound. Material D is a material of all wood-flour filler. In several types of apparatus tests, materials A and B have similar strengths whereas there is a substantial difference in their notched Izod values. On the other hand material D is notably weaker in the apparatus tests and correspondingly weak in the notched Izod strength.

Going to a comparison of material A and material C we find that these two materials are somewhat similar in notched Izod but dissimilar in apparatus strength.

Study of the data reveals discrepancies of one kind or another in any attempt to relate any single test on bars to apparatus strength. The increasing blow test, used widely in Europe, appears to offer no measurable advantage either with or without notch over the single-blow notched bar test, and of course the unnotched results show the same wide spread of values which normally occur when using this type of specimen. The increasing blow unnotched Izod, for example, shows material D to be strong whereas all apparatus tests and field data show it to be relatively weak. Both Charpy and Izod unnotched indicate a considerable difference between materials A and B which is not shown by apparatus tests.

TABLE IV.—STRENGTH RELATIONS OF THREE COMMERCIAL THERMOPLASTICS.

Material	Standard A.S.T.M. Notched-Bar Izod, ft-lb.	Unnotched-Bar Izod (See Table III), ft-lb.	Increasing Blow Notched-Bar Izod, in-lb.	Increasing Blow Unnotched-Bar Izod, in-lb.	Unnotched-Bar Charpy, ft-lb.	Flexural Strength, lb. per sq. in.	Modulus of Elasticity, lb. per sq. in.
A.....	0.144	0.509	1.10	5.75	0.634	6 400	487 000
B.....	0.342	0.917	2.20	8.86	1.292	5 900	271 500
C.....	0.125	1.720	1.00	16.6	2.523	13 850	....

No apparatus strength test results are available for the thermoplastics. Table IV gives the strength relations of three commercial thermoplastics as indicated by various tests on $\frac{1}{2}$ by $\frac{1}{2}$-in. bars. Thermoplastic A is a compression molding cellulose acetate, thermoplastic B is an extrusion molding cellulose acetate, and thermoplastic C is methacrylate resin. The relative strengths of the acetates are shown to be the same by any of the impact tests. In the flexural test, while the ultimate strength is of the same order, the relative stiffness, as indicated by the modulus values, is approximately in inverse ratio to the impact values. No such consistency is shown when comparing acetates and methacrylate. Although the notched Izod value of these particular methacrylate specimens is low, the unnotched values are unusually high. Which of these tests is of greater significance can only be determined by laboratory and field experience with the molded product.

Significance of a Group of Tests:

It is apparent from the above data that the impact breakage of plastic parts of all materials is far too complex a problem for solution by any single impact strength test. The data indicate, however, that at least one, and possibly more, combinations of tests can be depended upon to predict apparatus strength.

Under certain limiting circumstances, as already shown, a single test such as the notched Izod, or a combination of two tests would probably suffice. For broad applications, however, it appears that a combination of three strength tests on test bars will satisfactorily predict resistance to impact breakage of molded parts.

In choosing such a combination one is naturally governed by two considerations, namely, engineering sufficiency and economy. From these viewpoints, the choice becomes reduced to some combinations involving the notched and unnotched Izod, the Charpy, and flexural strength.

Since the present authors have some preference for the Izod over the Charpy, a combination of notched-bar Izod, unnotched-bar Izod, and flexural strength was tentatively chosen. Data covering about 25 batches of commercial phenolics from two sources show that in every case where all three tests on test bars gave high values, the apparatus molded from the same material was of high strength. Likewise in every case where one or more tests gave low values the product was relatively weak. The data are given in Table V.

TABLE V.—RELATION BETWEEN NOTCHED-BAR IZOD, UNNOTCHED-BAR IZOD, FLEXURAL STRENGTH AND APPARATUS MOLDED FROM THE SAME MATERIAL.

Materials represent commercial phenolics from two sources.

For convenience in analyzing the following data, a "good" material was considered to have a flexural strength of 10,000 lb. per sq. in., a notched-bar Izod strength of 0.150 ft-lb., and an unnotched-bar Izod strength of 0.675 ft-lb.

Material	Flexural Strength, lb. per sq. in.	Notched-Bar Izod, ft-lb.	Unnotched-Bar Izod, ft-lb.	Strength of Telephone Apparatus as Determined by Height of Fall Necessary to Break, in.
1......	Condition (A) 11 900	All three tests good 0.189	0.735	65
2......	Condition (B) 10 930	1 test poor 0.150	0.662	53
3......	Condition (C) 9 900	2 tests poor 0.235	0.584	45
4......	Condition (D) 9 700	3 tests poor 0.140	0.619	30

TABLE VI.—SHOWING THE NECESSITY FOR MOLDING CONTROL IN ATTEMPTING TO CORRELATE THE MECHANICAL STRENGTH TESTS ON TEST SPECIMENS WITH STRENGTH OF MOLDED TELEPHONE APPARATUS.

Material	Flexural Strength, lb. per sq. in.	Notched-Bar Izod, ft-lb.	Unnotched-Bar Izod, ft-lb.	Strength of Telephone Apparatus as Determined by Height of Fall Necessary to Break, in.
1..........	11 100	0.153	0.662	40.8
2..........	10 830	0.166	0.649	36.2
3..........	10 950	0.159	0.727	45.2
4..........	11 320	0 164	0 704	43.2
5..........	10 660	0.157	0.707	51.0
6..........	10 930	0.150	0.662	53.3
7..........	11 550	0.156	0.692	41.3
8..........	11 310	0.159	0.714	42.0
9..........	11 230	0.159	0.659	45.5
10..........	9 790	0.152	0.703	44.0

TABLE VII.—SHOWING THE SPREAD IN TEST RESULTS OF A PHENOL PLASTIC, UREA PLASTIC AND CELLULOSE ACETATE COMPOUND TESTED IN VARIOUS WAYS.

	Standard A.S.T.M. Flexural Strength Test, lb. per sq. in.[a]	Standard A.S.T.M. Notched Izod Strength Test, ft-lb.[b]	Unnotched Izod Impact Strength Test, ft-lb.[c]
Phenol Plastic...	10 670	0.186	0.633
	10 990	0.188	0.651
	11 250	0.189	0.656
	11 280	0.191	0.661
	11 290	0.193	0.674
		0.193	0.691
		0.194	0.692
		0.195	0.697
		0.198	0.709
		0.199	0.732
Urea Plastic......	9 450	0.145	0.565
	9 580	0.149	0.621
	12 750	0.154	0.625
	12 870	0.158	0.630
	13 030	0.160	0.661
		0.164	0.677
		0.164	0.714
		0.168	0.719
		0.175	0.727
		0.178	0.748
Cellulose Acetate.	3 780	0.156	0.292
	4 000	0.168	0.307
	7 810	0.171	0.343
	8 190	0.172	0.441
	8 230	0.173	0.483
		0.182	0.529
		0.186	0.652
		0.189	0.700
		0.203	0.746
		0.208	0.922

[a] Five bars molded from same material.

[b] Ten bars molded from same material.

[c] Ten bars molded from same material. (Standard A.S.T.M. bar, except unnotched.)

In compiling the data for Table V extremely careful control of molding conditions was exercised so that the molded product was truly representative of the raw material. The data in Table VI illustrate the importance of the close control. In the latter case the material used consisted of 10 batches of a particular commercial phenolic compound, and the molding conditions were allowed to vary through the widest commercial limits. In spite of the fact that the impact tests on the raw material indicate a reasonably consistent material, the strength of the molded product is highly variable.

Variation in Results:

In Table VII are shown data on phenol plastic, urea plastic and cellulose acetate when tested in various ways. It will be noted that the phenols are most consistent, the ureas next and the acetates poorest. This wide variability in test results on acetate is quite obviously independent of test methods. A background of testing this material for some ten years has created the impression among us that the general physical characteristics, including some properties not within the scope of this paper such as cold flow and "unmolding" (reverting to granular form), are substantially influenced by factors which are unknown, at least for the present.

Standard Methods of Test:

The standard methods of test for resistance to impact and flexural strength (Methods D 48 – 37)[4] were used throughout this investigation except as follows:

In measuring the flexural strength of soft thermoplastics where the specimen did not break, the maximum load was taken as the failure point.

For the increasing blow notched and unnotched Izod tests a ½ ft-lb. machine was used except in the case of the high-strength materials where the standard 2 ft-lb. machine was used.

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[4] Standard Methods of Testing Molded Materials Used for Electrical Insulation (D 48 – 27), 1937 Supplement to Book of A.S.T.M. Standards, p. 232.

DISCUSSION

Mr. Gerald H. Mains[1] (*presented in written form*).—Messrs. Burns and Werring are to be congratulated on this interesting and comprehensive paper which covers a field in which there have been little published data.

I was particularly interested in their conclusion that one single type of impact test would not serve to evaluate plastic materials from a performance standpoint. From the experience of our laboratory this is true not only where the molded sample and commercial product are not the same type piece, but even where the tests are made on the molded part itself. For testing certain molded phenolic laminated parts we have used the A.S.T.M. impact machine with a modified holder for an unnotched Izod type test perpendicular to the laminations, measuring total energy of break. We also used an increasing increment test on the same machine and found that certain pieces showed a pronounced surface crack under the increasing increment test at one-third to one-half the impact value required to completely break the piece. The presence of such a surface crack, of course, means an unsatisfactory piece no matter how high the ultimate breaking strength. In this case the increasing increment test enabled us to locate and correct defects that the total energy test did not pick out. On the other hand, we have had applications where the standard notched Izod impact test correlated better with service conditions than any other.

There is one point that might possibly be added to the discussion of size of test specimens (on pages 41 and 42 of the paper). The A.S.T.M. standard method permits the use of specimens of widths narrower than $\frac{1}{2}$ in., to be built up in a pile-up approaching $\frac{1}{2}$-in. width and tested edgewise. A.S.T.M. Methods D 256 – 34 T[2] call for impact values to be reported in terms of foot-pounds per inch of notch whether a single or composite specimen is used. In view of this it would seem desirable to modify the statement in the present paper (page 42, first paragraph): "It is therefore permissible only to state absorbed energy in terms of a standard specimen." It might be well to mention that all of the values given in the paper are in terms of the $\frac{1}{2}$ by $\frac{1}{2}$-in. specimen and not in foot-pounds per inch of notch.

The authors refer to the work of Committee D-9, showing that a preconditioning treatment of 48 hr. at 50 C. was sufficient to erase previous atmospheric history of the specimen and to obtain reproducible results. It might also be mentioned that under this drying condition, actual values for the various grades of phenolic laminated and phenol plastic studied were only 5 to 15 per cent lower than values under average conditions of approximately 40 per cent relative humidity. In other words, for many materials there would be little use for an average conditioning treatment in addition to the preconditioning procedure described.

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[2] Tentative Methods of Testing Electrical Insulating Materials for Resistance to Impact (D 256 – 34 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 995 (1934); also 1937 Book of A.S.T.M. Tentative Standards, p. 1023.

MR. W. A. ZINZOW[3] (*presented in written form*).—The authors are indeed fortunate in being connected with an organization which enables them to follow so closely the history of the material from the unfinished form through the molding process into the numerous applications as well as the subsequent history of the finished product. Few laboratories are in a position which enables them to correlate material test data with field service results under such widely varying conditions and at the same time have such complete information about these varying service conditions. The excellent correlation between laboratory test data and field service results reflects the quality of the laboratory data.

TABLE I.

	A END	B END
Energy to break, ft-lb.	0.157	0.176
Energy to carry away broken end of test piece, ft-lb.	0.104	0.096
	0.103	0.097
	0.092	0.103
	0.095	0.096
	0.101	0.098
	0.095	0.102
Averages......................	0.0983	0.0987

There is one factor which enters into the determinations of impact strength of plastic materials which the authors of this paper have not discussed but of which they are well aware. That is the effect of the weight of the broken pieces on the amount of energy absorbed from the moving pendulum. The energy values to break test specimen for some of the materials discussed run between 0.10 and 0.20 ft-lb. Since the broken pieces are carried away with the pendulum at an appreciable velocity, a certain amount of the energy lost by the pendulum may be used up in imparting this velocity to the broken pieces. This was called to our attention when we were determining the effect of prolonged heat treating at high temperatures on the impact strength of some materials. In spite of the fact that some pieces had practically no strength, which was evidenced by the fact that they could be crumbled easily between the fingers, the impact values obtained never dropped below certain minimum values. To check this point we then broke some standard materials using the 2 ft-lb. Izod tester described in this paper. After breaking, the broken end of the test specimen was set in the position it had before the test; the pendulum was then released and some surprising results were obtained. The accompanying Table I shows that the pendulum lost about 0.0985 ft-lb. of energy in carrying away the broken end of the test specimen which originally had an impact strength of 0.166 ft-lb. energy to break. Thus, on a single test specimen the energy required to carry the piece away was more than half of the total energy required to break the piece and carry the end away. What does this mean now? We believe it means that the difference between a 0.15 ft-lb. material and a 0.25 ft-lb. material may be greater than those values would indicate. We believe that this may be a rather important refinement which needs to be applied to the impact testing of plastic materials, particularly those of low impact strength.

MR. MAURICE H. BIGELOW.[4]—In considering the data presented in this paper, a question arises in our minds which we think should be presented at this time. In Committee D-20 on Plastics we are actively engaged in considering the proper method of testing plastics for resistance to shock strength. We are not interested only in the phenolic type of molding compound but all molding compounds, including some twenty different types. The question that arises in the impact testing of plastics is the previous history of the molding compound itself, particularly with respect to its water content. We have reason to believe that in some molding compounds there is an absorption of water by the molding compound and that this absorbed water has an effect upon the plasticity of those materials. In softening the material, as the absorption of water inevitably does, there is a difference in the strength characteristics of the molded bar from those of the original molding compound prior to the water absorption. It seems, therefore, imperative that in supplying a molding compound to a laboratory for impact testing, some consideration be given as to how long the material was stored and how it was stored as particularly relating to temperature and humidity conditions.

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[3] Chief Physicist, Bakelite Corp., Bloomfield, N. J.

[4] Technical Representative, Plaskon Co., Inc., Toledo, Ohio.

MR. GORDON M. KLINE.[5]—I have a couple of questions that I should like to ask the authors. In Fig. 3, the effect on impact strength of an increase in the moisture content seems to be to increase the impact strength more rapidly on the unnotched specimens than on the notched specimens up to 0.20 per cent moisture content. Above this percentage just the opposite effect takes place, the notched specimens increasing in impact strength more rapidly than the unnotched. I wonder whether the authors had any explanation of this behavior. Of course it is very curious to see the notched specimen having approximately the same impact strength as the unnotched at the higher moisture content. One wonders what would happen if one went beyond the 0.35 per cent moisture content and whether the notched specimens would have been stronger than the unnotched.

Then, in Table IV some data are given on the impact strength of cellulose acetate and methacrylate plastics. With all due recognition of the merits of methacrylate resins, we know that the cellulose acetate plastic under normal conditions is a remarkably tough material, and one wonders why in this test the methacrylate resin would show such superior impact strength. Of course, the obvious answer is that the conditioning at 50 C. (122 F.) is responsible for it, but for a plastic such as cellulose acetate which contains quite an appreciable quantity of water under ordinary conditions, it does not seem quite representative to test it in the dry condition as it would be after 48 hr. at 50 C. I wonder whether the authors have any information on the equilibrium relations as to moisture content of the cellulose acetate plastic after 48 hr. at 50 C. That is, to what relative humidity does the test condition correspond? I think it would probably be much lower than the 20 per cent relative humidity which is shown in Fig. 2 to be the usual indoor relative humidity during the winter months.

MR. WALTER W. WERRING.[6]—Mr. Mains' reference to the use of a repeated blow test to determine the first crack in a laminated plastic is very interesting. This would appear to be particularly effective for tests of laminated materials struck flat-wise, as the "leaf spring" characteristics of the laminations might cause great differences in total energy to break, though the initial cracking value might be similar for different materials. We have encountered problems of a similar nature in specification tests for molded plastic parts in which electrical conductors were embedded. A single blow pendulum test would yield a high value of which the larger part would be the breaking off or pulling out of the wires and consequently variations in the strength of the plastic would be masked. Determination of the energy to cause the first crack was found to be a very useful criterion of the strength of the plastic. Our chief difficulty with such tests has been the definition of "the first perceptible crack."

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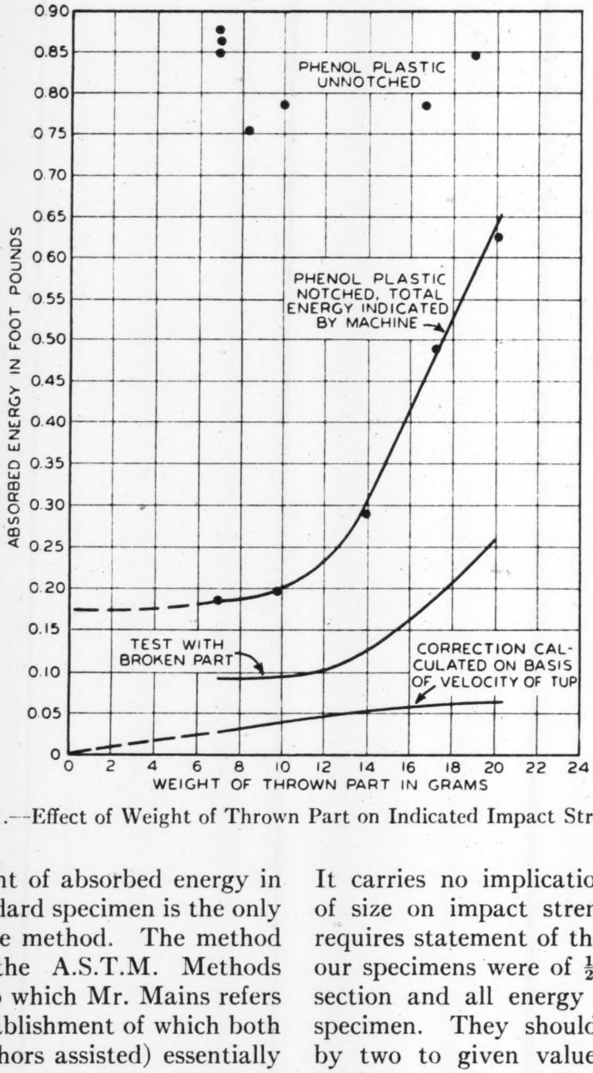

FIG. 1.—Effect of Weight of Thrown Part on Indicated Impact Strength.

The statement of absorbed energy in terms of a standard specimen is the only strictly accurate method. The method described in the A.S.T.M. Methods D 256 – 34 T to which Mr. Mains refers (and in the establishment of which both he and the authors assisted) essentially does this because, though it calls for a report in terms of foot-pounds per inch of notch, it at the same time requires statement of the individual molded width of the specimens actually broken in the test. This method of reporting gives a convenient approximate comparison between data on materials which are tested in say $\frac{1}{2}$-in. thickness and others whose maximum thickness may be $\frac{1}{8}$ in It carries no implication of the effect of size on impact strength and hence requires statement of the test size. All our specimens were of $\frac{1}{2}$ by $\frac{1}{2}$-in. cross-section and all energy values are per specimen. They should be multiplied by two to given values according to Methods D 256 – 34 T.

Mr. Zinzow referred to the energy

used in driving off the broken end of the specimen and, in the example which he cites, it can easily be a large part of the indicated energy. However, high-speed moving pictures show that in many cases the specimen seems to carry itself off. In high-speed pictures of photoelastic studies the transparent specimen, which was only slowly stressed by the pressure screw, flew off at high speed due to its own resilience. Even with the pendulum blow, the hard rubber left the vise at a higher speed than the motion of the pendulum. It would appear that in materials of high resilience a correction for the energy in the thrown piece would approach zero but that in one of low resilience there is need of some correction.

Some time ago I made an effort to obtain some quantitative information on this subject. I arranged to vary the weight of the thrown ends from the same lot of specimens by adding various amounts of lead wire evenly distributed about their centers of gravity and then breaking the specimens in the regular way. The accompanying Fig. 1 shows data from this test.

It will be noted that the data plotted at the top of the figure for the unnotched phenol plastic and the next lower solid curve below it for the notched phenol plastic illustrate the effect of resilience already mentioned. The unnotched specimen can carry additional weight without requiring additional energy from the pendulum. This is not always true in the case of the notched specimen for which we would obviously have to make a correction of 0.4 ft-lb. were the thrown end as heavy as 20 g.

The results obtained by setting the broken end back in position and driving it off with the pendulum are plotted as the next lower solid curve. This does not show the same increase in indicated energy with increase in weight that the notched specimen does. The reason for this is presumably that the sort of motion imparted to the thrown end depends, among other things, upon the way it is restrained at the bottom when struck. The gyrations of the specimens shown in the moving pictures were different for different materials. That these rotary gyrations may require considerable energy is indicated by comparing the higher curves with the lowest curve which shows the energy calculated to be required for straight translation of the thrown end at the velocity at which the tup is moving after breaking the specimen.

This figure is not to indicate the value of the correction but to show how complicated the establishment of a proper correction would be. I agree with Mr. Zinzow that in the case of the very fragile material used in his example some quite large correction is probably necessary. I am at a loss to say what it should be for the complete range of materials as it must vary from the maximum of Mr. Zinzow's example all the way to zero. I can only suggest that in particular cases a test of weighted specimens such as I have described may, by the shape of the curve, indicate the magnitude of the correction.

However, we are fortunate in that the usual commercial plastics are of similar weights and have sufficient resilience not to require corrections of large or different magnitudes. As illustrated by the curve for the notched phenol plastic, the weight of the thrown end may be increased to 10 g. without greatly affecting the indicated energy. This is an increase in weight of approximately 50 per cent which is a great change in an organic plastic material.

Mr. Bigelow's comments bear on a subject which is very important but is often neglected—the necessity for representative test specimens. In metals the test specimen may be machined from a portion of the material actually used but in plastics the production of test bars is a separate operation. All the implications of this difference are sometimes not appreciated. Sampling of a plastic for test is really not completed with the selection of a quantity of powder from the bulk shipment. It is not completed until completion of the molding of the test bars and if the test bars are to be representative of the product all intermediate steps in storage, handling, etc., must be representative. This situation is well recognized in the case of electrical properties, as absorption of moisture by the powder may result in poor product from material which is good on test or *vice versa*. It is just as important though less recognized in the case of mechanical properties. In our view the material that is being sampled is not the *molding powder* but the *molded product*.

In reply to Mr. Kline's questions on Fig. 3, I have no definite explanation of why the changes in moisture content have different effects upon notched and un-notched strengths. That it does have this different effect is part of the basis for our belief that the notched test is the most significant single test. The curve indicates that a decrease in moisture content in the range of field conditions causes little change in the un-notched strength and also in transverse strength but makes a large difference in the notched strength. As we know that such a change produces a considerable increase in field breakage, the notched test appears to parallel field experience with this material. The mechanism by which the moisture change operates to this end is rather obscure. However, the figure does not, as he suggests, show the notched and un-notched strengths to be the same. The two curves are drawn to different scales. Although our data do not extend far enough to be certain about it, I do not think that the curves will cross. The effect of moisture increase apparently is to reduce the notch effect and therefore the notched strength could hardly exceed the strength of the material without a notch.

With regard to the questions on the data of Table IV, it might be well to point out that our intention has been to limit ourselves to the subject of impact testing and particular materials are mentioned only for purposes of illustration. Mr. Kline's comment that cellulose acetate is generally considered tougher than the methacrylate resins is substantially true. However both materials cover a wide and overlapping range. The tough acetates usually thought of are either sheet materials or soft extrusion molding compounds whereas the compound referred to was a hard one, and compression molded. It will be recognized that compression molding a $\frac{1}{2}$-in. thick section of cellulose acetate is a very unfavorable condition for this material, more unfavorable in fact than the conditioning. With regard to more favorable conditioning, which seems to be suggested, the data at hand indicate that conditioning at a humidity such as 20 per cent to a reproducible condition eliminating the effects of past history on impact strength would hardly be a practicable method of test. In the case of phenol plastic, conditioning by oven drying requires 48 hr., for acetate it requires 4 days which is rather objectionably long. Similar conditioning at 20 per cent relative humidity would for phenol plastic require from 12 to 28 days. The time required for cellulose acetate has not yet been determined but it is obvious that it will be so long as to have a place only in a research project. It is not known at this time to what equilibrium humidity condition 48 hr.

at 50 C. corresponds but we agree with Mr. Kline that it is lower than 20 per cent relative humidity.

MESSRS. ROBERT BURNS[7] and WALTER W. WERRING (*authors' closure by letter*). —The authors appreciate the interest in their paper displayed by those offering discussions. These valuable discussions and other informal comments received since the meeting have shown an increasing realization of the importance of the preparation of test specimens of plastics. We have long believed that much of the difficulty in obtaining data significant of the performance of plastic products may be traced to the difficulty of producing representative test specimens. It appears desirable to add here some additional information on our own methods.

In the production of most molded products, considerable flow of material occurs in the mold, yet the rectangular specimens necessary for test have usually been molded in a straight compression mold which for this shape of piece produces little if any flow of material. We all know that this difference alone invalidates the test results for many materials, and materials such as cellulose acetate can hardly be knit together at all without flow. Tests have been made of molding methods using various distributions of material in the mold but we have found the straight compression mold generally unsatisfactory. The result has been the development of a new type of mold which we have now used for a number of years.

We call this mold a "step mold," because it molds five test bars at one time in a single cavity in which the bars are arranged at different levels like steps. A single bar is at the bottom and two bars are arranged in steps above it at each side. A correspondingly stepped plunger completes the mold. A single charge of material is used and the material must flow sideways up the steps and through the thinning fin between bars as the mold closes. This mold, with appropriate time and temperature cycles, has been found to be more generally satisfactory for all materials than any method of which we know. However, if product molding is done by any special method such as by extrusion, the production of specimens should be by corresponding methods.

The notch is milled in the molded specimens by means of a special form milling cutter before conditioning. The notching must be done before conditioning so as to avoid any effect due to heating in the machining and also so that the bottom of the notch will be subjected to the conditioning. The moisture effect is to a large extent a surface one and the specimens after conditioning must be kept in a small dessicator right up to the moment of testing. Even laying out a lot of 10 specimens on the bench at the start of the test will, in humid weather, result in progressively higher values as the 10 specimens are tested. We cannot emphasize too strongly the need for careful and consistently representative methods in the preparation and handling of specimens if significant data are to be obtained.

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PRACTICAL APPLICATION OF THE NOTCHED-BAR IMPACT TEST

BY G. C. RIEGEL[1] AND F. F. VAUGHN[1]

SYNOPSIS

In our experience, the utility of the notched-bar impact test lies in its ability to detect and evaluate the effects of many factors upon which the quality and reliability of steel and iron products depend: namely,

I. Relative metal quality aside from that associated with dynamic loads.

II. The reliability of the quality of heat treatment.

III. The relative sensitivity to cleavage under conditions of suddenly applied stresses, or frequent overloading by gradually applied stresses.

Certain figures and data are submitted in verification of these premises.

For many years, we have accorded the notched-bar impact test a ranking position among the various mechanical tests relied upon for assurance of fulfillment of the physical properties required in the vital parts of machines such as we produce. We feel that the service performance of our products over this same period of years has justified and substantiated our faith in the ability of the notched-bar impact test to discriminate between materials of satisfactory and unsatisfactory "notch sensitivity." Furthermore, such discrimination could *not* have been accomplished through the medium of the usual methods of static tests which were made in conjunction with the notched-bar impact test.

In our experience, the utility of the notched-bar impact test lies in its ability to detect and evaluate the effects of many factors upon which the quality and reliability of steel and iron products depend: namely,

I. Relative metal quality aside from that associated with dynamic loads.

II. The reliability of the quality of heat treatment.

III. The relative sensitivity to cleavage under conditions of suddenly applied stresses, or frequent overloading by gradually applied stresses.

Without going into the details of the size and form of the specimen, and the shape of the notch, since these subjects have been quite extensively treated in the writings of others,[2, 3, 4] we determined, during experiments conducted in the period from 1927 to 1929, that at least two types of notched specimens were necessary to cover ferrous materials from the most brittle to the toughest employed.

The materials which break with a relatively low absorption of energy can be best examined, in our judgment, with the Charpy specimen shown in Fig. 1. The notch is not sufficiently sharp to make the spread of foot-pound values too narrow. The depth of the notch eliminates the influence of surface effects, which is not true of the same type of specimen with a shallow notch. The hole can be produced by drilling, thereby avoiding the introduction of undesirable transverse scratches.

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[1] Chief Metallurgist, and Assistant Chief Metallurgist, respectively, Caterpillar Tractor Co., Peoria, Ill.

[2] Max Moser, "Notched-Bar Tests," *Journal*, Am. Soc. Mechanical Engrs., Vol. I, No. 3, July-September, 1933, p. 105.

[3] S. L. Hoyt, "Notch-Bar Testing," *Metals and Alloys*, January, p. 5, February, p. 39, April, p. 102, and May, p. 140 (1936).

[4] "Notch-Bar Impact Testing," *Proceedings*, Manchester Assn. Engrs., October 29, 1937:

R. V. Southwell, "Impact Testing from a Physical Standpoint," p. 1.

L. W. Schuster, "Some Aspects of Notched Bar Test," p. 15.

Max Moser, "The Development and Present Position of Continental Research on Notched Bar Impact Test," p. 55.

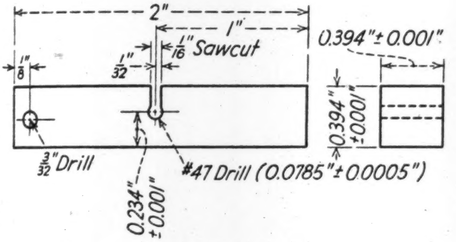

FIG. 1.—Charpy Type Impact Specimen. Saw cut must not strike side or bottom of drilled hole.

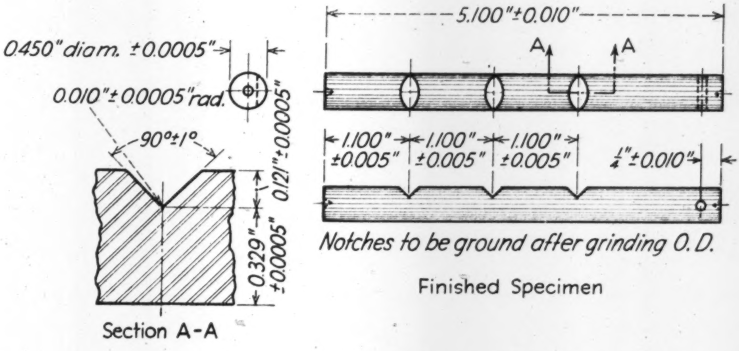

FIG. 2.—Izod Type Impact Specimen.

For materials which are considered tough—those which break with a relatively high absorption of energy—the specimen shown in Fig. 2 has best served our purpose for the following reasons:

1. It has a sharp notch, the sharpness of which is definitely known and controllable. (In experimenting with sharply-notched specimens, for example, Charpy, Izod, and tension impact, we found the foot-pound values profoundly influenced by the sharpness of the notch, as should be expected. The 90-deg. angle, with a 0.010-in. radius, was selected as being the sharpest notch of accurate reproducibility.)

2. The Izod specimen, as compared with the Charpy type, avoids energy absorption of an unknown amount at the point of percussion and fracture of tough materials, and likewise avoids the error due to pulling the specimen in compression and tension between the anvils.

3. It permits the use of a hardened cap at the point of percussion, thereby avoiding the error due to deformation by denting of tough material.

4. It provides an easy form for mechanical preparation of such commonly used materials as bolts, studs, cap screws, tie rods, deposited weld metals, and even the ends of broken tension specimens.

5. In addition to affording three tests on one specimen, it may be prepared for one or two breaks when material is limited.

Modification of Notched-Bar Test Specimens:

Supplementing the specimens already shown, we have used a specimen similar to Fig. 2 with no notch for very brittle materials, and with a 0.060-in. depth of notch for specimens taken from highly hardened material returned from service, which is only machinable by grinding. Such specimens can be initially cut, by a rubber disk, from the part and held under water while centers are brazed on for subsequent finishing by grinding. This has been of considerable value in determining the quality of the original heat treatment of the material.

For extremely brittle material, the test specimen requires no notch because the first suddenly applied stress induces brittle cleavage. For this reason, we have employed, since 1930, a 0.750-in. diameter ground specimen for tool steel, and a 1.125-in. diameter ground specimen, similar to Fig. 3, for cast iron.

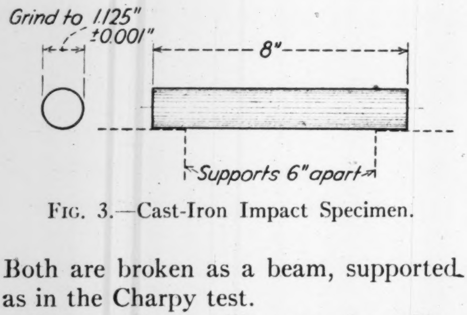

FIG. 3.—Cast-Iron Impact Specimen.

Both are broken as a beam, supported as in the Charpy test.

In our effort to illustrate the utility of the test as a means of evaluating the factors affecting the quality and reliability of steel products as previously enumerated, we shall consider each phase separately from the standpoint of our experience in the practical application of the test.

I. RELATIVE METAL QUALITY

In 1926, it became apparent to the management of our company, that the heat treatment and field performance of successive mill heats of steel of the same type or analysis, could not be adequately predicted from precisely conducted tension testing. It is now well known that two mill heats of the same typical chemical composition and almost duplicate tensile properties may possess strikingly different notched-bar impact values as shown in Table I,[5] which are actual results.

As an illustration of the field performance of a mill heat such as that which we have indicated as inferior, after a certain mill had made ten successive heats of steel which failed to meet our minimum Charpy requirements, we selected one as being the best of these heats, and 400 sets of gears were made from this heat and placed in our machines. For four successive years thereafter, gears from this lot failed under severe conditions of service, while other gears from heats which passed our minimum Charpy requirements gave satisfactory performance, free from service complaint.

TABLE I.

	Carbon, per cent	Manganese, per cent	Sulfur, per cent	Phosphorus, per cent	Silicon, per cent	Nickel, per cent
Mill heat A.....	0.46	0.61	0.031	0.016	0.20	3.57
Mill heat B.....	0.47	0.70	0.036	0.018	0.24	3.55

PHYSICAL PROPERTIES (DRAWN AT 425 F. FOR 1½ HR.)

	Rockwell Hardness, "C" Scale	Tensile Strength, lb. per sq. in.	Yield Point, lb. per sq. in.	Elongation in 2 in., per cent	Reduction of Area, per cent	Charpy Impact, ft-lb.	Tension Impact,[a] ft-lb.
Mill heat A (good)......	52	278 000	214 000	11.5	44.3	18.0	62
Mill heat B (poor).......	53	288 500	214 500	11.5	34.1	5.5	9

[a] Refer to notched specimen in Fig. 4.

During the 10-yr. period, 1928 to 1938, we have subjected 2737 mill heats of steel to notched-bar impact tests. Of these, 146 mill heats were not accepted.

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[5] G. C. Riegel, "Selection and Heat Treatment of Stee for 'Caterpillar' Tractors," *The Iron Age*, Vol. 131, No. 12, March 23, 1933, p. 468.

and many of those which were accepted had limited application due to their low response to the notched-bar impact test. This number of mill heats included both plain carbon and highly alloyed steel. Much of this material was applied to parts in which dynamic loading in service is not a part of the engineering consideration. Nevertheless, the freedom from failures is closely parallel to the classification made on the basis of the notched-bar test.

On this basis of classification, through the cooperation of steel producers, it has been possible, during this same period of time, to increase by gradual steps the hardness of our tractor track links from 302 Brinell to 444 Brinell, with the expectation of deriving considerable increase in wear resistance, without any reduction in the minimum Charpy values, thereby maintaining the same reliability in service.

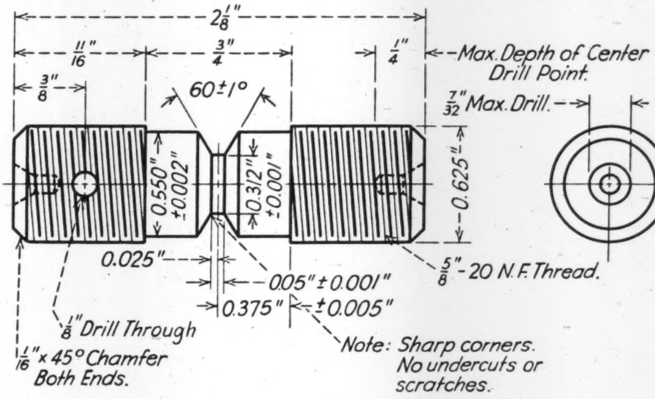


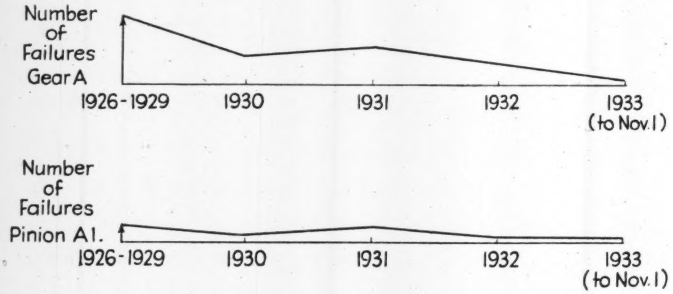
FIG. 4.—Notched Tension-Impact Specimen.

II. RELIABILITY OF QUALITY OF HEAT TREATMENT

For at least 15 yr., the present company and one of its predecessors has specified notch toughness as one of the requirements of heat-treated cold-finished steel used for studs, bolts, and tie rods. The original method of testing and acceptance was based upon results of the nick-bend or notch-fracture test. When early attempts were made to transfer this nick-bend acceptance test to some corresponding Izod value, it was found that some of the specimens fell below 25 ft-lb. The immediate result was the effort on the part of the steel source to raise this minimum to 30 ft-lb. This was accomplished by improved practice in heat treatment. Subsequently, it was possible to increase the minimum to 35 ft-lb. through closer control of heat treatment. These requirements were based upon an alloy steel.

At this point, after a thorough investigation of the response to the Izod test made on mill heats of alloy and carbon steel of several types, with the objective of maintaining the minimum of 35 ft-lb. Izod and a minimum of 80,000 lb. per sq. in. yield strength (0.2 per cent offset) in the heat-treated condition, it was found that good quality, properly hardened and tempered carbon steel would more than fulfill these requirements. Thereupon, the selection of mill heats of carbon steel upon the basis of response to heat treatment as measured by the notched-bar impact test made it possible to raise the minimum Izod requirement to 50 ft-lb. This experience has embraced over 18,000 tons of bars. Subsequent experience has shown that further refinement of heat treatment will make possible a minimum Izod requirement of 70 ft-lb.

Breakage of certain steel castings on tractors had become quite irritating about seven years ago. Investigation of service failures revealed irregular and non-uniform heat treatment by the vendor. This was proved by the low and erratic Charpy impact values, ranging from 2.5 to 12 ft-lb. The same castings, when properly heat treated, gave Charpy values from 8 to 17 ft-lb. Accordingly, the specification was revised to require the better heat treatment and a minimum Charpy value of 8 ft-lb. for acceptance. As a result, service failures from this cause are practically unknown, and such castings are used on many thousands of machines in the field.



Yearly average for the period 1926 - 1929 has been used since only the total experience for this period is known.

FIG. 5.—Comparison of Annual Failures of Tractor Final-Drive Gears and Pinions of S.A.E. No. 2345 Steel.

III. RELATIVE SENSITIVITY TO CLEAVAGE

Twelve years ago, when there was scant knowledge of the relative sensitivity to cleavage, or "separation fracture," as Moser called it,[4] of the different types of steel, it was thought possible to replace, with impunity, one type of gear steel with another. Steels having equivalent properties of tensility and hardness at normal temperature were assumed to have an equivalent response to service requirements. This imperfect knowledge took no account of the relative behavior of "notch sensitivity" of steels at different temperatures.

For example, on a few machines, S.A.E. No. 2345 steel was replaced by S.A.E. No. 5150 type steel in certain gears, on the assumption that the latter type would respond more readily and uniformly to heat treatment and would show slightly better wear resistance at the same hardness. It also had the advantage of better machinability and hence should permit greater accuracy in the tooth form.

After these machines were placed in service, notably at extremely cold temperature, gear breakage became alarming. The notched-bar test at normal temperature, was immediately called into play for examining this difference in behavior between the two types of steel. Not having an impact testing machine available, it took several blows with a hand hammer, accompanied by considerable bending, to break the nickel steel, as compared with a single blow and no bending of the chromium steel, both having the same degree of hardness.

Subsequent Charpy impact tests made on these two materials, at gear hardness, proved not only their wide difference in "notch sensitivity" at room temperature (17 ft-lb. for the nickel steel, and less than 5 ft-lb. for the chromium steel) but tests conducted at − 20 F. showed very slight loss in foot-pound values for the nickel steel, whereas the chromium steel lost as much as 50 per cent.

As practical evidence of improvement in service performance, based upon the selection of steel by an acceptance test including a minimum Charpy value, the chart in Fig. 5 shows graphically the progressive reduction in service failures of gears made from the same type of steel so tested. The types of gear failures encountered here are of two kinds: teeth broken out as by a sudden blow with no evidence of progression, and teeth which fail progressively under repeated overstressing.

TABLE II.—CHARPY SPECIMENS QUENCHED AND TEMPERED TO ROCKWELL HARDNESS, "C" SCALE, OF 53.

	SINGLE WIDTH SPECIMENS, FT-LB.	DOUBLE WIDTH SPECIMENS, FT-LB.
Mill heat C.	7.5	6.75
Mill heat D.	14.5	30.0

When we find a heat of steel which is close to the minimum foot-pound value with our single width specimen, as shown in Fig. 1, we believe that a more critical examination of the steel is necessary by the use of the intensified notch effect of a specimen similar to that shown in Fig. 1, but of *double* the *width*.

If, under this more severe test, the heat of steel gives *double* the foot-pound values of the single width specimens, our suspicions of relative sensitivity to brittle cleavage are allayed. If the foot-pound values of the *double* width specimens are less than *double* that of the single width specimens, the heat is not accepted for the purpose intended.

The examples shown in Table II will serve to illustrate the discrimination afforded by this method on certain mill heats of S.A.E. No. 2345 type steel.

Effect of Surface Conditions:

One of the factors frequently overlooked in making a notched-bar impact test with specimens notched before heat-treatment is the pronounced influence of surface conditions on the "notch sensitivity" or cleavage strength of the steel. For example, a Charpy specimen of S.A.E. No. 2345 steel, heat treated in the usual slightly *oxidizing* atmosphere, to a Rockwell hardness, "C" scale, of 52, gave a value of 14 ft-lb. The same steel, heat treated to the same hardness, but in a furnace atmosphere which was slightly *carburizing*, having produced a carburized depth of but 0.0015 in., gave a value of 2 ft-lb. Conversely, the same material, heat-treated in the same manner to produce the equivalent hardness, with an atmosphere which *decarburized* the surface to a depth of 0.0015 in., gave a value of 19 ft-lb.

Another instance of difference in degree of "notch sensitivity," due to stress concentration, is illustrated by the difference in Charpy impact values of gear steel specimens notched *before* and *after* heat treatment which was conducted in the same furnace and at the same time, using a slightly oxidizing atmosphere and producing after tempering a Rockwell hardness, "C" scale, range of 50 to 52. The specimens notched *before* heat treatment gave an average value of 4 ft-lb., whereas those notched *after* heat treatment, gave an average value of 6 ft-lb.

Conclusions

1. The notched-bar impact test is useful in evaluating the quality of ferrous metals, even though the material is not called upon to resist impact stresses in service.

2. The notched-bar impact test is a most discriminating proof of the quality of heat treatment of ferrous metals.

3. The notched-bar impact test predicts the behavior of ferrous metals under conditions which produce brittle cleavage.

DISCUSSION

Mr. Arthur B. Wilder[1] (*presented in written form*).—The results described in this paper are of interest to those engaged in fundamental research, because of the practical applications which have resulted from the use of the notched-bar impact test. H. F. Moore and I have found the impact test of value in a study of the influence of grain size on the properties of metals. The tensile strength may remain the same with variable grain size in certain materials but impact values vary according to the change in grain size.

It would be of interest to know of the method employed for meeting the tolerance limits specified for the specimen described in Fig. 2. The metallurgical microscope has been used for observing the shape of tool and notch with marked success. A magnification of 100 appears to be satisfactory.

The authors have indicated that the width of the specimen in Fig. 1 may be doubled with the result of doubling the impact value. This information is very useful in impact testing and, as they have indicated, applies only to acceptable material.

Mr. W. H. Bruckner[2] (*presented in written form*).—It is indeed gratifying to have the authors' correlation of the impact values of various materials with the service behavior of the materials. The authors have an exceptionally good opportunity to make such observations in their plant due to the rigid control of materials and heat treatment which are exercised. The control of material by specification and close inspection is costly but pays dividends in enhanced integrity of the product manufactured as shown in the curves of Fig. 5.

It is important that the two sentences on page 63 be emphasized by the authors. "Much of this material was applied to parts in which dynamic loading in service is not a part of the engineering consideration. Nevertheless, the freedom from failures is closely parallel to the classification made on the basis of the notched-bar test." This authoritative statement, backed by years of checking test values with service behavior, should do much to dispel the doubt that has arisen as to the value of the impact test for other than impact service.

For the manufacturing consumer of steel products it would be ideal to have all the properties high; tensile strength, yield, impact strength along with ductility at the lowest cost for the material. It is, therefore, of considerable interest to note that the authors have been able to get high properties with lower cost for the material (carbon steel *versus* alloy steel) by means of inspection tests which select the desirable heats.

With regard to the details of the paper I should like to ask why the $\frac{3}{32}$-in. drilled hole is provided in the Charpy specimen in Fig. 1. Also, have the authors made any tests of half-width Charpy specimens for materials they suspected as being undesirable and for which they usually make tests on the double-width sample?

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[1] Assistant Professor of Metallurgical Engineering, University of Illinois, Urbana, Ill.

[2] Research Associate in Metallurgical Engineering, Engineering Experiment Station, University of Illinois, Urbana, Ill.

MR. G. H. WRIGHT[3] (*presented in written form*).—Referring to Table II, I would like to ask if mill heats C and D were heat-treated in the same manner and if so was any special heat treatment applied to improve heat C. Our experience has shown that reheat-treatment of these notch-sensitive steels has been beneficial when we quenched from the draw-back temperature, followed by a slow reheat to approximately 300 C. and then slowly cooled.

MR. A. B. KINZEL:[4]—I should like to extend my compliments to the authors for the thorough presentation of the correlation of service and impact performance that we have long been seeking. The correlation is shown very clearly, certainly as far as the particular applications cited are concerned. There are two illustrations in the paper on which I should like to comment: First, the substitution of carbon steel for alloy steel in bolts and studs. With the application of modern knowledge in metallurgy, particularly the advent of grain size control and the *S*-curve, this can be done. However, the author might mention that such substitution is restricted to relatively small-size bars, and that in the larger sizes the heat treatments are no longer effective, so that to get strength the alloy steels are necessary. Second, a comparison between the nickel-chromium steel and straight chromium steel is made. This comparison was certainly valid at the time the test was made and was representative of much of the material delivered under these classifications. However, since then, the whole story of *S*-curves, grain size and special deoxidation has come into the picture. I do not wish to provoke a controversy as to whether or not a similar conclusion would be valid today, but certainly we must review the situation and I strongly suspect that the difference between the two steels would be of a very different order of magnitude if the steels were made and heat-treated under modern practice. Mr. Riegel rightly points out that those tests were made 12 yr. ago and the above comments are merely intended to call attention to the changes that have taken place. They do not in any way affect the validity of the authors' main conclusions, and I know that he is fully aware of the limitations of the carbon steels and the improvement in some of the alloy steels.

MR. L. B. TUCKERMAN.[5]—This paper, on first reading, made an excellent impression on me. Here at last, it seemed, there was presented definite evidence of correlation of impact tests in the laboratory with service records; evidence which is all too scanty in the publications on impact testing. Detailed reading, however, left a feeling of some disappointment. The authors have not given the evidence in as definite a form as I could have wished. I found myself wanting more definite evidence before I could be fully convinced. I have listed a few of the points on which I would have liked to have further evidence.

Figure 5 is convincing evidence of the decrease of service failures which followed the specification of minimum Charpy values for the steel used in gear A. This is very definite but I found myself wondering whether it was not merely a case of *post hoc, ergo propter hoc*, and whether between 1926 and 1933 there was no change made in the design of gear A or pinion A-1, or no other measures taken to remedy the situation. You all remember the very thorough study of rear axle gear failures reported by Almen and Boegehold[6] from which they drew the conclusion that "Due to their influence on stress concentration, variations in design and manufacture, . . . have a materially greater effect on gear life than do variations in materials and heat treatments."

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[3] Works Metallurgist, Schenectady Works Laboratory, General Electric Co., Schenectady, N. Y.

[4] Chief Metallurgist, Union Carbon and Carbide Research Laboratories, New York City.

[5] Assistant Chief, Division of Mechanics and Sound, National Bureau of Standards, Washington, D. C.

The authors do not make it clear whether mill heat B, of Table I, was the one they selected as being the best of ten heats which failed to meet their minimum Charpy requirements and from which the four hundred sets of gears were made.

Conclusion 1, states: "The notched bar impact test is useful in evaluating the quality of ferrous metals, even though the material is not called upon to resist impact stresses in service." The only statement in the body of the paper upon which this conclusion can be based seems to be the following: "Much of this material was applied to parts in which dynamic loading in service is not a part of the engineering consideration. Nevertheless the freedom from failures is closely parallel to the classification made on the basis of the notched-bar test." It would add much to the value of the paper if records of service failures were given of parts in which not merely "dynamic loading in service is not a part of the engineering consideration," but in which the nature of the service was such as to exclude the possibility of accidental dynamic loading contributing to the failure. The paper is a useful addition to our knowledge of impact testing but it would have been more useful if the laboratory tests and the service records had been more definitely correlated.

MR. G. C. RIEGEL[7] (*authors' closure*).—I certainly wish to thank the many discussers of this paper for their interest. Mr. Bruckner spoke about the use of half-width specimens as compared with the whole width, but the geometry of the half-width specimen is wrong for the notched-bar test when you attempt that correlation, and hence we abandoned it, as explained by other writers, notably Hoyt's articles in *Metals and Alloys* in 1936 as referred to in the paper.

Referring to the last paragraph of Mr. Bruckner's discussion, the $\frac{3}{32}$-in. hole in the Charpy specimen is used for convenience in handling the specimens in the quenching and tempering treatments. By means of wires placed through the holes, they are supported and kept separated during these operations.

The other remarks by Mr. Bruckner in confirmation of the authors' results are the more appreciated by the authors in view of the fact that he is very familiar with the test methods and manufacturing practices discussed.

Mr. Tuckerman spoke about some of the conclusions having rather scant background of evidence, which I must admit; we condensed it greatly, but the gear failures that were recorded represented several things, first, that every pinion and every gear was marked with a steel identity; over the period of experience covered, something like twenty thousand machines had been built and the heat treatments were constant, in so far as we knew how to make them so. They were all run in the "Hump" furnaces by the critical change curve and the design of the parts was constant. The mill heat referred to in Table I did happen to be the particular mill heat from which the four hundred gears were chosen for use, because at that time mill heat "B" was considered to be the nearest to passable under our specifications. There were other mill heats of a lower order than the one marked poor.

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[6] J. O. Almen and A. L. Boegehold, "Rear Axle Gears: Factors Which Influence Their Life," *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part II, p. 99 (1935).

[7] Chief Metallurgist, Caterpillar Tractor Co., Peoria, Ill.

I have some data that I should be glad to give to anyone who inquires about mill heats which have been heat treated to attain 295,000 lb. per sq. in. tensile strength. The remainder of the properties in tension are practically identical in every respect, that is, having an elongation of 12 per cent, reduction of area of 45 per cent, and whose areas under the stress-strain diagram would be almost alike. In the one instance, the Charpy values might range from 2.0 to 4.0 ft-lb. and in the other instance they would fall between 12.0 and 16.0 ft-lb. as we should expect. This indicates the futility of attempting to predict notch touchness from the area under the stress-strain curve.

In reply to the last paragraph of Mr. Tuckerman's discussion, the authors meant to convey by the expression—"Much of this material was applied to parts in which dynamic loading in service is not a part of engineering consideration"—that such parts were not engaged in the direct transmission of power, nor were they subjected directly to contacts such as blows in which energy must be suddenly absorbed.

I appreciate very much the statement made by Mr. Kinzel. The remarks are quite to the point in the present comparison between the two types of steel: namely, S.A.E. No. 5150 type and S.A.E. No. 2345 type, one made under the practice today and the other ten years ago. Yet, some of the work published by A. J. Herzig and R. M. Parke in their article entitled "Laboratory Investigation of Low-Temperature Impact Properties of Some S.A.E. Steels" in *Metals and Alloys*, April, 1938, Vol. 9, No. 4, page 90, shows that the departure between those two steels is still pretty wide when measured for notch sensitivity.

With respect to his remarks regarding the size limitations for the use of carbon steel in studs, bolts, and tie rods, we may say the maximum size in these applications in carbon steel has been $1\frac{1}{4}$ in. in diameter.

Replying to Mr. G. H. Wright, the method of heat treatment was identical for both mill heats: namely, C and D. The hardness requirements of the parts for which these mill heats were intended would not permit the use of draw temperatures as high as suggested by Mr. Wright.

Replying to Mr. Wilder's question, the radius at the bottom of the notch and the tools used in producing the same are checked by the use of a Bausch & Lomb projector at approximately 100 diameters magnification.

THE USE OF THE CHARPY TEST AS A METHOD OF EVALUATING TOUGHNESS ADJACENT TO WELDS

By Walter H. Bruckner[1]

Synopsis

A series of steels with varied chemical contents were welded with a single welding bead placed on the surface of ½-in. thick flat plates of the steels. The heat-disturbed area adjacent to the weld deposit was surveyed for hardness, grain size and microstructure. The area of microscopic extent where maximum hardness and grain size occurred adjacent to the single-bead weld was physically magnified by synthesizing the microstructure by means of a "weld-quench" heat treatment. The latter treatment produces in a half-size Charpy bar practically the same maximum hardness, grain size, and microstructure as is found adjacent to the single-bead welds.

Keyhole-notch Charpy impact tests on the bars of "weld-quenched" material were compared with impact tests made on the parent steels in the "as-received" condition. The comparison indicates that when the chemistry of steels for welding is suitably chosen, there is little or no loss in toughness adjacent to the weld. For such steels the rate of transformation of the austenite is sufficiently rapid to form high-temperature products. Where steels were encountered which were sensitive to the welding quench, a low value of toughness was obtained, coupled with a delayed or low rate of transformation of austenite.

Preheating tests made on a series of the steels sensitive to the welding quench indicated that in general the transformation of austenite can be shifted to higher temperatures by preheating, with the result that soft, high-temperature products are obtained in the microstructure. The determination of toughness of the heat-disturbed area in such preheated welding tests is suggested as a valuable contribution to our knowledge of welding of the "tricky" steels.

The "weld-quench" test is successful in separating welding steels into two categories: (1) steels insensitive to the welding quench and therefore easily welded without preheat or other special handling, and (2) steels sensitive to the welding quench such that special care, preheat or preheat and subsequent heat-treatment are required for successful welding.

The test is therefore recommended as a low-cost method for use in the development of additional steels of easy weldability, for checking heats of steel for weldability before rolling to welding plate size, or for matching heats of steel already in successful welding use in the consumers' plants. The purchaser of steels for welding has with the "weld-quench" test a rational basis for specifying his requirements in lieu of the previously used addition to the specification "and the steel shall be of weldable quality."

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As introduction to the present paper, reference is made to a lengthy article by the author in the *Journal* of the American Welding Society for October, 1937, with the title "The Heat Effect in Welding—A Review of the Literature to January 1937."

The literature which was searched for the above review indicates that research activity has recently been intensified in order to define a test or method of testing which will determine prior to welding whether a material is weldable. It is evident that special attention has been paid to the maximum hardness produced in the heat-disturbed area in welded joints, since experience in welding the steels indicates that this region is usually the site of cracks if they occur at all. Another reason for hardness criteria is that hardness is the most easily obtainable property of the cross-sectioned welded joint.

A limit has been placed on maximum hardness for standard single-bead welding tests as shown in the investigations reported by W. G. Theisinger **(1)**[2] and W. L. Warner **(2)**. This limiting value of maximum hardness has been set by various authors in another way, by specifying that martensite shall not appear in the microstructure next to the weld.

Factors other than maximum hardness have been injected into the consideration of weldability; these are the bend ductility and toughness of the heat-affected zone. It has been shown by A. Fry **(3)** and Musatti and Reggiori **(4)** that a steel which offers no difficulty in arc welding and does not harden excessively has a high notch-impact value. Hardenability and toughness are therefore related. Further proof of the relationship is given in the work of Meunier and Rosenthal **(5)** in which it is shown that the notch-impact values of single-bead welds decrease as the hardenability rises with increasing carbon content. The relationship between hardenability and notch-impact value is, however, not exactly parallel in the single-bead weld tests of the latter authors. The hardness increases gradually with carbon content while the impact value decreases rapidly. The bend ductility *versus* carbon shows a rapid drop in the curves beyond 0.33 per cent carbon.

Investigations of welded joints with the Chevenard micromechanical testing machine **(6)** also indicate that the relationship between shear ductility and tensile strength of small specimens taken progressively through the joint is not an exact one. For a series of steels tested, the reduction of ductility of the heat-affected zone with increase in strength does not appear entirely consistent.

We also have an example in the tests reported for gas-welded steels by Kleiner and Bossert **(7)** for actual welds and by Kinzel **(8)** for furnace-treated samples to duplicate various regions of the heat-affected zone. The authors find critically low values of impact resistance with a fairly low hardenability in the region adjacent to the fusion line due to the peculiar structure developed there. The determination of progressive hardness values across the welded joint is therefore seen to be inadequate for establishing a criterion for the properties possessed by the heat-disturbed zone. This conclusion is not an original one, but appears in the literature. The report by J. H. Critchett **(9)** is an excellent presentation of this view and many of the steels investigated by the present author are discussed from the large amount of data tabulated from the welding literature by Mr. Critchett's committee.

In order to establish a quantitative

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[2] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p 93.

criterion of the weldability of steels, a program of research was initiated by the Bureau of Engineering, Navy Department, and carried out at the Naval Research Laboratory. The research was projected under the title "The Weldability of Iron Alloys" with the objective of a "weld-quench" test to be established for determining the toughness of the heat-affected area adjacent to welds. The weld-quench test was initially conceived as an impact test on a small bar of the parent steel which had undergone a heat treatment to duplicate the large grain, maximum hardness region of the heat-disturbed area adjacent to welds.

TABLE I.—CHEMICAL ANALYSES AND HISTORY OF MATERIALS INVESTIGATED FOR WELDABILITY.

Item	Carbon, per cent	Manganese, per cent	Silicon, per cent	Nickel, per cent	Copper, per cent	Molybdenum, per cent	Chromium, per cent	Vanadium, per cent	Columbium, per cent	Thickness, in.[a]	History
1	0.17	0.41	0.17								Silicon-killed, hot rolled steels Carbon series
2	0.25	0.43	0.20								
3	0.35	0.68	0.23								
4	0.44	0.65	0.24							$\frac{3}{8}$	
5	0.21	0.38	0.003								Rimmed heat, hot rolled steels
6	0.27	0.47	0.002								
9	0.24	0.48	0.23								Hot rolled steels Manganese series
10	0.27	0.74	0.21								
11	0.29	1.06	0.25								
13	0.26	0.46	0.005								Hot rolled steels Silicon series
14	0.28	0.54	0.058								
15	0.25	0.43	0.20								
17	0.22	0.52	0.20			0.19					Hot rolled steels—laboratory heats rolled in commercial mill—Molybdenum series
18	0.22	0.47	0.18			0.50					
19	0.20	0.50	0.17			0.79					
20	0.24	0.46	0.001								Hot rolled boiler steels
21	0.25	0.43	0.19								
22	0.18	1.40	0.21					0.12			Hot rolled manganese-vanadium steel
23	0.15	0.44	0.20		0.75	0.17					Hot rolled—laboratory heat-rolled according to commercial practice
24	0.28	0.56	0.27	3.57							Hot rolled — Nickel steels Carbon series for 3½ per cent nickel
25	0.19	0.40	0.18	3.63						$\frac{3}{4}$	Rough forged
26	0.23	0.48	0.19	3.55						$\frac{3}{4}$	
27	0.06	0.35	0.002	1.95	0.94						Hot rolled low alloy, high tension welding steels
28	0.21	0.70	0.27	1.97	1.08						
29	0.028	0.19		2.08							Hot rolled nickel ingot iron
30	0.09	0.72	0.047	0.96	1.34	0.11					Hot rolled, normalized and levelled low alloy, high tension
31	0.24	0.74	0.012	0.76	1.48	0.16					
32	0.12	0.75	0.48				0.34				Hot rolled, low alloy welding steel
33	0.11		0.19			0.51	5.85				Hot rolled, low chromium steel modified with molybdenum
34	0.065		0.22			0.51	5.66		0.55		
35	0.27	0.69	0.20	2.31							Nickel steel, hot rolled
B	0.26	0.64	0.20								Grade M hull plate 1½ in. thick — Hot rolled steels for mass effect tests
36	0.27	0.69	0.21	2.30							Nickel steel 1¾ in. thick
51	0.31	0.55	0.42								Cast steels. Casting 6 by 9 by ¾ in. Heated after casting to 1600 to 1650 F. for 1 hr., air cooled. Reheated to 1200 F., held 1 hr. and furnace cooled Surface of plates were sand blasted
52	0.14	0.54	0.54								
53	0.26	0.63	0.48								
54	0.43	0.61	0.44								
55	0.25	1.26	0.64								
56	0.40	0.73	0.48			0.62					

[a] All the above steels were supplied in 6 in. wide plate, ½ in. thick except where noted otherwise as to thickness.

MATERIALS

A series of structural steels varying in content of carbon, manganese, silicon, molybdenum, nickel and chromium were assembled. The series represents plain carbon and low-alloy steels, a few of the latter being generally considered as especially suitable for welding The analyses are given in Table I. In general the steels are of commercial grade rolled out in regular mill practice. Except where noted otherwise the steels were $\frac{1}{2}$ in. thick plate. Several analyses of cast steels were included in order to obtain comparisons of castings with similar rolled steel compositions.

A lot of grade EA class 2 heavily coated electrodes was obtained in $\frac{3}{16}$-in. size for use in all of the welding tests.

TEST METHODS

Welding tests were made on 3 by 6-in. sections of the $\frac{1}{2}$ in. thick plates of rolled steels. The plates were surface ground before welding to remove scale and surface imperfections. A bead of weld metal was deposited on the surface of each plate at room temperature with the electrode on reversed polarity and carrying 180 to 190 amp. direct current at 25 to 27 v. The speed of the electrode was maintained constant at 6 in. per min. with the plate to be welded stationary, the bead of weld metal being deposited on the 6-in. length in the middle of the 3-in. width of the plate.

A large lathe was fitted out to move the electrode horizontally at the desired speed while the electrode holder was free to be manipulated manually in the vertical direction for constant arc conditions maintained by a skilled welder. The plates to be welded were held in contact with a $\frac{3}{4}$-in. thick steel table for electrical contact only, and were not in any sense restrained from expanding or contracting.

A $\frac{1}{2}$ in. thick section was cut from all the welded plates transverse to the weld bead and at a distance of $3\frac{1}{2}$ in. from the end of the plate where the weld was started. The sections were polished as for microexamination, etched with 1 per cent Nital and subjected to a hardness survey with a Vickers type hardness testing machine. A 10-kg. load was used on the diamond pyramid with which hardness tests were made 0.05 in. below the welded face of the plate at intervals of 0.02 in. in the heat-disturbed area adjacent to the weld, and at intervals of 0.05 in. outside in the undisturbed parent metal (see Fig. 1). From the hardness surveys a value of maximum hardness and a hardness contour were obtainable.

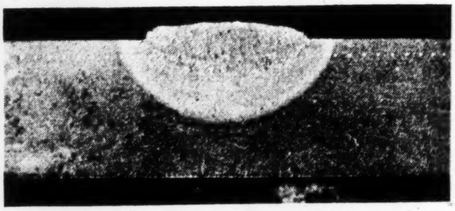


FIG. 1.—Method of Making Hardness Survey on Single Bead Weld.

The single-bead welded plates were also surveyed under the microscope to determine the size of the largest grains adjacent to the weld bead and the microstructural characteristics of the heat-disturbed area in the region of maximum hardness.

Weld-Quench Tests:

A quenching procedure was then developed with which it was possible to produce a sample 2.0 by 0.404 by 0.207 in. with practically the same hardness, grain size and microstructure as in the area of maximum hardness for the corresponding single-bead welds. This heat treatment is called the weld-quench test and consisted of heating the above sample in a furnace at 1350 to 1365 C. supplied with atmosphere control and maintained slightly carburizing. The samples were held in the furnace for $1\frac{1}{2}$ min. and immediately transferred to a bath of molten sodium hydroxide at a temperature of 530 C. They were held in the latter bath for 18 sec. and transferred immediately to a second bath of sodium hydroxide with a 40 molecular per cent addition of potassium hydroxide at 310 C. where they were held for 24 sec. and air cooled. The samples were then surface ground to remove 0.005 in. all over; to finish to a 2.0 by 0.394 by 0.197-in., half-size Charpy bar which was prepared with a keyhole notch and broken in impact.

For heat treating the samples for the weld-quench test, a holder was made and attached to each sample in order to obviate the use of tongs. This holder consisted of a 1-in. length of $\frac{1}{8}$-in. steel wire threaded on both ends. One end of the wire was screwed into a threaded hole in one end of the sample to a depth of $1\frac{1}{2}$ threads; the other end of the wire was screwed into the end of a $\frac{1}{4}$-in. diameter rod 24 in. long.

It was determined from previous tests that it required $1\frac{1}{2}$ min. to heat the half-size Charpy bar just to the furnace temperature of 1350 to 1365 C. All samples were therefore accurately timed with a stop watch as to time in the furnace and in the two quenching baths. Three samples of each steel were weld-quenched according to the above schedule in order to obtain an average value for the three closely checking values.

Two tests of half-size Charpy impact resistance were made on the as-received steel of each grade in order to compare values before and after the weld-quench test. The impact values in all tests reported in this paper were made on the Amsler machine with the pendulum in the same initial position; thus the striking velocity and initial energy were the same for the entire series of tests.

Thermal Cycle of Single-Bead Welds:

In the development of the weld-quench test it was necessary to determine the thermal cycle of the region of maximum hardness in the heat-disturbed area of the single-bead welds. To do this a study was made of the contour of the fusion line and heat-disturbed area of several plates which had been single-bead welded according to the previously described welding schedule. A 2-mm. hole was drilled in other plates of the same steels, the center of the hole being at a depth of 0.08 in. from the welded face and a distance of 0.01 to 0.02 in. from where the fusion line would be when the plate was welded. The bead of a No. 40 B & S. gage platinum-platinum-rhodium thermocouple was spot welded into the center of the flat bottom of the 2-mm. hole, the wires being led off to the side of the plate through a 0.04-in. wide milled slot 0.08 in. deep. The slot was cut perpendicular to the line of the weld bead. The center of the slot was in line with the center of the 2-mm. hole. The thermocouple wires were placed in the slot between a lower and upper layer of thin mica sheet and insulated from each other by means of powdered magnesium oxide. The oxide powder was also sprinkled on the spot-welded thermocouple bead. Both the drilled hole and slot were filled with a metal plug and strip respectively which were spot welded into the plate in order to approximate the original continuity of the plate.

With the above assembly, the temperature-time curve was obtained for a distance of 0.01 and 0.02 in., approximately, from the fusion line under the same conditions of welding as obtained

TABLE II.—SUMMARY OF HARDNESS TESTS ON SINGLE-BEAD WELDS.

Welding Conditions.—Grade EA, class 2, $\frac{3}{16}$ in. heavy coated electrode, 180 to 190 amp., 25 to 27 v., speed 6 in. per min.

Hardness Testing Conditions.—Vicker's type hardness testing machine, 10-kg. load. Indents 0.05 in. from top of plate (welded face), 0.02 in. apart in transition zone and 0.05 in. apart outside.

Item	Thickness, in.	Maximum Hardness in Transition Zone	Parent Metal Hardness	Hardness Increase	
				Vickers Brinell Units	Per Cent
1		193	135	58	42
2		221	148	73	49
3		266	169	97	57
4		(340)[a]	210	130	62
5		203	143	60	42
6		243	150	93	62
9		224	143	81	57
10		287	172	115	67
11		312	181	131	72
13		230	150	80	53
14		237	157	80	51
15		227	151	76	50
17		230	153	77	50
18		254	177	77	44
19		287	186	101	54
20		218	143	75	53
21		243	148	95	64
22		312	197	115	58
23		237	160	77	48
24		503	212	291	137
25		345	174	171	98
26		413	183	230	126
27		195	155	40	26
28		405	206	199	97
29		170	126	44	35
30		254	200	54	27
31		397	218	179	82
32		221	152	69	45
33		437	152	285	188
34		383	171	212	124
35		383	195	188	96
MASS EFFECT TESTS					
B	1½	282	146	136	93
	1	292	"	146	100
	¾	266	"	120	82
	½	237	"	91	64
	¼	212	"	66	45
36	1¾	503	187	316	169
	1½	493	"	306	164
	1	483	"	296	158
	¾	455	"	268	143
	½	446	"	259	138
	¼	282	"	95	51
CAST STEELS					
51		270 (Avg. 276)	154	122	79
		282			
52		215 (Avg. 215)	130	85	65
		215			
53		282 (Avg. 287)	147	140	95
		292			
54		421 (Avg. 425)	179	246	138
		429			
55		405 (Avg. 401)	177	220	125
		397			
56		613 (Avg. 620)	204	416	204
		627			

[a] Calculated, plate less than ½ in. thick.

for the single-bead welds on which hardness surveys were made. The points on the curve were obtained from the projection of a movie film which contained a record of the millivoltmeter reading and elapsed time of a stop watch. The photographic record was made by the movie camera at a speed of 16 frames per second.

Welding Tests with Preheat Treatment:

In order to establish the prior data for the weld-quench test, it was necessary to make a survey of comparative weldability on the basis of hardness increase, microstructure, etc., for the various steels in single-bead welding tests at room temperature. Some of the steels appeared to give little promise of easy weldability as judged by the standardized weld-quench test but it was known that some of the steels were being successfully welded by preheating. The aim, therefore, in making preheating tests was to establish the comparative prior data with respect to grain size, maximum hardness and microstructure which would serve as a basis for developing modified weld-quench tests. The modified tests were conceived as a classification of the "tricky" steels on the basis of the preheat required to give a standard value of toughness or loss of original toughness in the heat-affected area.

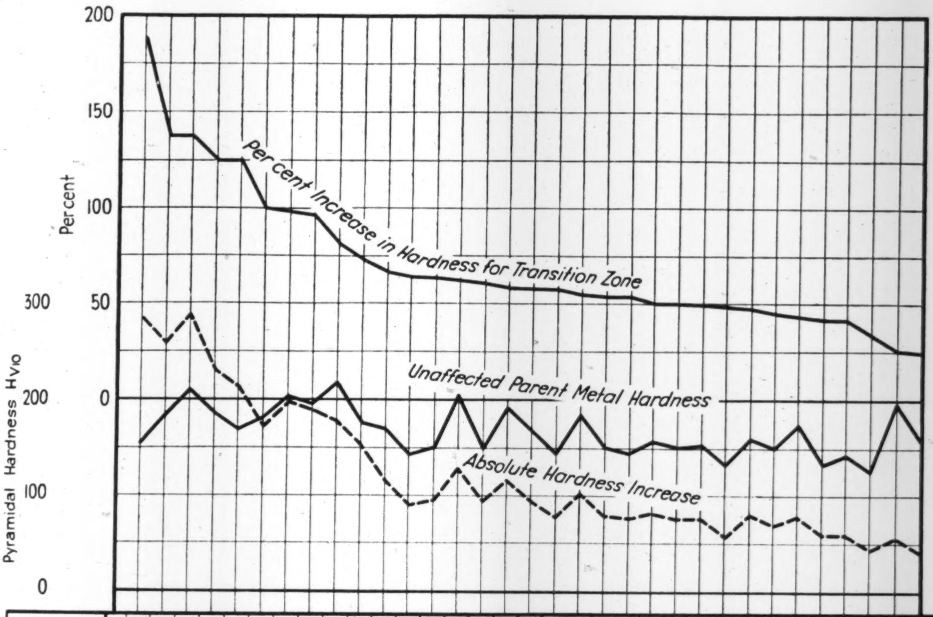
The preheating tests were carried out by preheating to 300, 500 and 700 F. (150, 260 and 370 C.) a series of steel plates which gave low weld-quench values. Plates of the same size and thickness as in previous welding tests were heated in a furnace to the desired temperature and quickly transferred to a sheet of asbestos placed over the ¾-in. steel welding table. Quick-closing clamps provided the electrical contact.

and welding was started at the same speed of 6 in. per min. and arc conditions as previously. Sections ½ in. wide were cut from the single-bead welds which were then polished, etched, and surveyed as before for Vickers hardness. The modified weld-quench tests have not as yet been developed from the data supplied by the preheating tests, but the author hopes to carry this phase of the problem to its conclusion in the near future

TEST DATA

A summary of the hardness surveys on all single-bead welded plates is given in Table II. In the table are also given the results of a series of tests on increasing mass, or thickness of plate for two grades of steel.

The values in Table II for welding tests on the cast steels are the averages of duplicate tests on 3 by 6 by ¾ in. thick castings, which were welded on the sandblasted surface after cleaning



Item	33	36	24	26	34	25	28	35	31	11	10	B	21	4	6	22	3	9	19	13	20	14	15	17	2	23	32	18	1	5	29	30	27
Carbon	0.11	0.27	0.28	0.23	0.066	0.19	0.21	0.27	0.24	0.29	0.27	0.26	0.25	0.44	0.27	0.18	0.35	0.24	0.20	0.26	0.24	0.28	0.25	0.22	0.25	0.15	0.12	0.22	0.17	0.21	0.028	0.09	0.06
Manganese	–	0.69	0.56	0.48	–	0.40	0.70	0.69	0.74	1.06	0.74	0.64	0.43	0.65	0.47	1.40	0.68	0.48	0.50	0.46	0.46	0.54	0.43	0.52	0.43	0.44	0.75	0.47	0.41	0.38	0.19	0.72	0.35
Silicon	0.19	0.21	0.27	0.19	0.22	0.18	0.27	0.20	0.012	0.25	0.21	0.20	0.19	0.24	0.002	0.21	0.23	0.23	0.17	0.005	0.001	0.058	0.20	0.20	0.20	0.20	0.48	0.18	0.17	0.003	–	0.047	0.002
Nickel		2.38	3.57	3.55		3.63	1.97	2.31	0.76																						2.08	0.96	1.95
Copper							1.08		1.48																	0.75						1.34	0.94
Molybdenum	0.51				0.51				0.16										0.79					0.19		0.17		0.50				0.11	
Vanadium																0.12																	
Columbium					0.55																												
Chromium	5.85				5.66																						0.34						

Note - Dash (–) above indicates presence of element but no analysis

FIG. 2.—Comparison of Hardness Values in Single-Bead Welds.

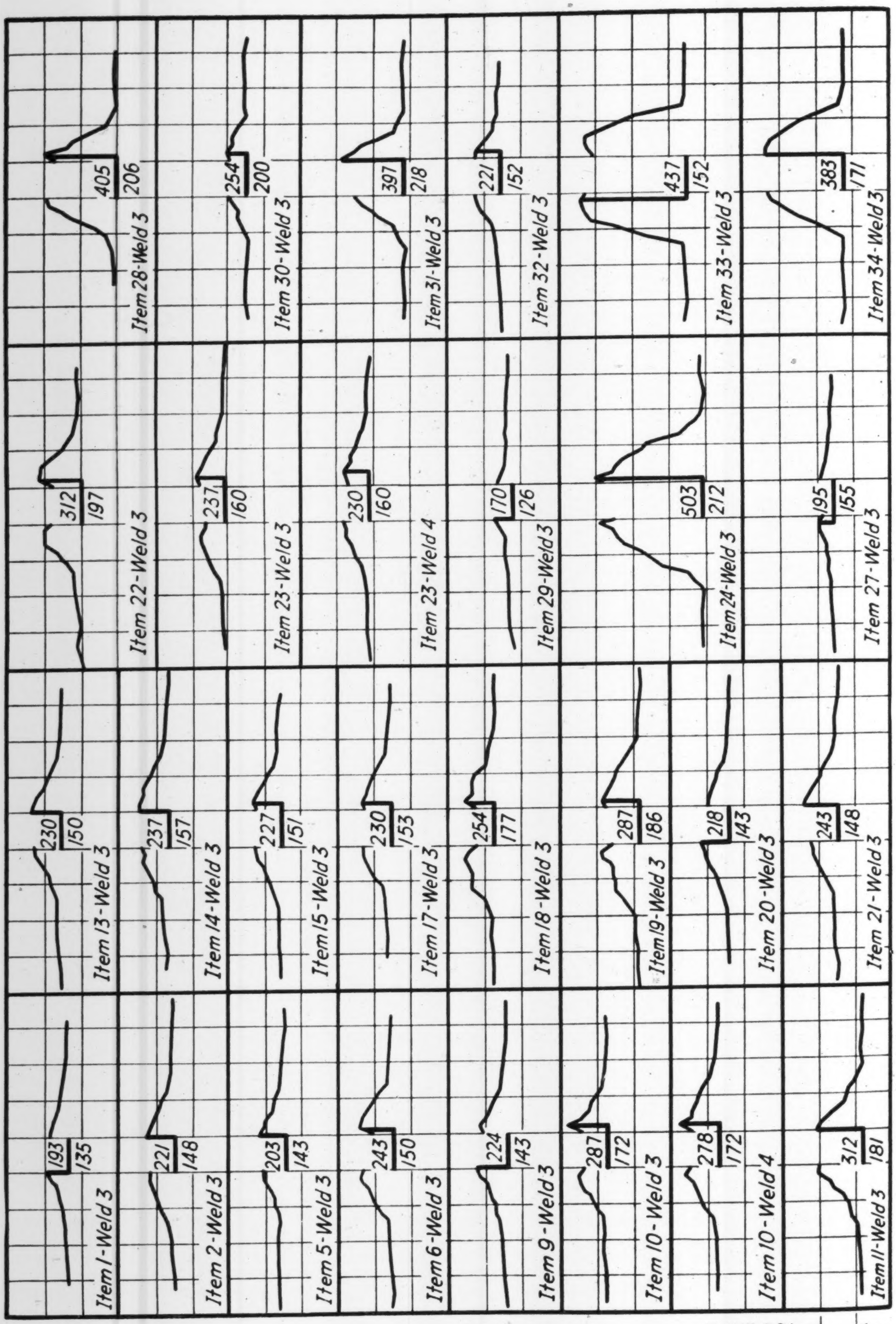


Fig. 3.—Hardness Contours Obtained from Single Bead Welding Tests.

with carbon tetrachloride. The hardness maxima for the cast steels are therefore not directly comparable with the ½ in. thick plates of rolled steel.

The results of the hardness surveys made on the ½-in. plates of rolled steels are compared in Fig. 2 which is arranged in the order of highest to lowest value of per cent hardenability. It will be noted that at the right-hand end of this figure desirable combinations of high parent metal hardness (high ultimate strength) and low hardenability are encountered for certain steels which have been developed by the industry especially for fool-proof welding. It may be noted in connection with this figure that several investigators have advocated a maximum hardenability of 100 units for welding steels.

A few of the hardness contours plotted from the hardness surveys made on the single-bead welds are shown in Fig. 3. An example of the reproducibility obtainable in the welding tests is given in the contours of duplicate tests on items 10 and 23. It will be noted that with each contour is given the maximum hardness value with a line showing where it occurs on the contour. The average parent metal hardness value is given below the value of maximum hardness.

The effect of alloying elements in rais-

TABLE III.—IMPACT AND HARDNESS DETERMINATIONS IN WELD-QUENCH TESTS ON ROLLED STEELS.

Item	Chemical Composition, per cent									Impact			Hardness		
	Carbon	Manganese	Silica	Nickel	Copper	Molybdenum	Chromium	Vanadium	Columbium	As Received Material (Average of Two Tests), ft-lb.	Weld-Quench Samples (Average of Three Tests), ft-lb.	Percentage of Original Impact After Weld-Quench Treatment	Maximum Hardness for Heat-Affected Area in Single-Bead Welding Tests	Average Hardness of 3 Weld-Quench Samples with 3 Indents Each Sample	Percentage Attainment in Weld-Quench of Maximum Hardness in Welding Tests
1...	0.17	0.41	0.17	.....	.....	.....	.....	.....	.....	16	18	112	193	208	108
2...	0.25	0.43	0.20	.....	.....	.....	.....	.....	.....	16	15	94	221	224	101
3...	0.35	0.68	0.23	.....	.....	.....	.....	.....	.....	19	13	68	266	260	98
4...	0.44	0.65	0.24	.....	.....	.....	.....	.....	.....	10	2½	25	(340)[a]	392	115
5...	0.21	0.38	0.003	.....	.....	.....	.....	.....	.....	17	12½	74	203	233	115
6...	0.27	0.47	0.002	.....	.....	.....	.....	.....	.....	16	15	94	243	242	100
9...	0.24	0.48	0.23	.....	.....	.....	.....	.....	.....	9	9	100	224	234	105
10...	0.27	0.74	0.21	.....	.....	.....	.....	.....	.....	19	9	47	287	253	88
11...	0.29	1.06	0.25	.....	.....	.....	.....	.....	.....	22	7	32	312	323	104
13...	0.26	0.46	0.005	.....	.....	.....	.....	.....	.....	14	16	114	230	237	103
14...	0.28	0.54	0.058	.....	.....	.....	.....	.....	.....	13	12	92	237	250	106
15...	0.25	0.43	0.20	.....	.....	.....	.....	.....	.....	16	14	88	227	242	107
17...	0.22	0.52	0.20	.....	.....	0.19	.....	.....	.....	25	28	112	230	221	97
18...	0.22	0.47	0.18	.....	.....	0.50	.....	.....	.....	24	25	104	254	246	97
19...	0.20	0.50	0.17	.....	.....	0.79	.....	.....	.....	25	20	80	287	267	94
20...	0.24	0.46	0.001	.....	.....	.....	.....	.....	.....	16	15	94	218	232	106
21...	0.25	0.43	0.19	.....	.....	.....	.....	.....	.....	16	12	75	243	241	100
22...	0.18	1.40	0.21	.....	.....	.....	.....	0.12	.....	13	7½	58	312	377	121
23...	0.15	0.44	0.20	.....	0.75	0.17	.....	.....	.....	23	26	113	237	229	97
24...	0.28	0.56	0.27	3.57	.....	.....	.....	.....	.....	19	2	11	503	450	89
25...	0.19	0.40	0.18	3.63	.....	.....	.....	.....	.....	19	4	21	345	384	111
26...	0.23	0.48	0.19	3.55	.....	.....	.....	.....	.....	20	3½	18	413	415	100
27...	0.06	0.35	0.002	1.95	0.94	.....	.....	.....	.....	30	30	100	195	205	105
28...	0.21	0.70	0.27	1.97	1.08	.....	.....	.....	.....	18	3	17	405	404	100
29...	0.028	0.19	.....	2.08	.....	.....	.....	.....	.....	31	27	87	170	163	96
30...	0.09	0.72	0.047	0.96	1.34	0.11	.....	.....	.....	24	30	125	254	237	94
31...	0.24	0.74	0.012	0.76	1.48	0.16	.....	.....	.....	19	6	32	397	372	94
32...	0.12	0.75	0.48	.....	.....	.....	0.34	.....	.....	30	27	90	221	225	102
33...	0.11	.....	0.19	.....	.....	0.51	5.85	.....	.....	36	14	39	437	411	94
34...	0.065	.....	0.22	.....	.....	0.51	5.66	.....	0.55	37	7	19	383	369	97
35...	0.27	0.69	0.20	2.31	.....	.....	.....	.....	.....	15	2½	17	383	432	113
36...	0.27	0.69	0.21	2.30	.....	.....	.....	.....	.....	21	3	14	446	414	93
B...	0.26	0.64	0.20	.....	.....	.....	.....	.....	.....	19	15	79	237	237	100

[a] Calculated, plate less than ½ in. thick.

ing the maximum hardness adjacent to the weld is shown in Fig. 4. The values were obtained from the hardness surveys of the single-bead welds.

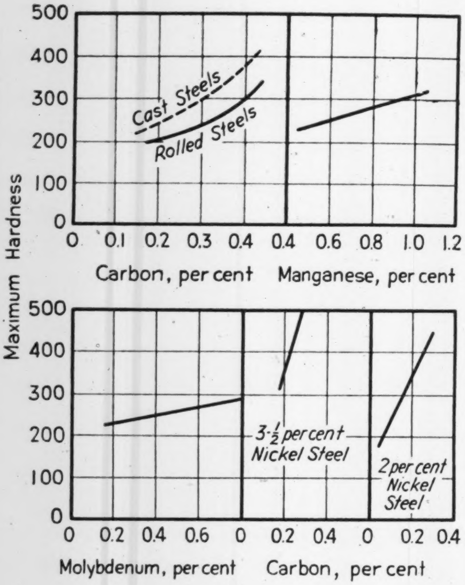


FIG. 4.—Maximum Hardness *versus* Composition for Transition Zone.

TABLE IV.—IMPACT AND HARDNESS DETERMINATIONS IN WELD-QUENCH TESTS ON CAST STEELS.

Item	Impact			Hardness, Weld-Quench (average of four samples)
	As Received (average of five tests)	Weld-Quench (average of four tests)	Percentage of Original Impact	
51.....	8.6	3½	41	289
52.....	17.2	11	64	228
53.....	10.6	8½	76	265
54.....	4.0	1½	38	329
55.....	11.5	4	35	331
56.....	Casting excessively porous, no weld-quench tests made			

NOTE.—The "as received" condition of the castings is described in Table I.

Weld-Quench Tests:

The data summarizing the weld-quench tests made on all the steels are given in Tables III and IV. In Table III are given the results of impact tests made on the half-size, Charpy keyhole notched bars of the rolled steels before and after the weld-quench heat treatment. The effect upon impact values of the weld-quench treatment is shown in the third column. The fourth column of Table III reports the maximum hardness values of Table II for single-bead welds which are to be compared with the fifth column on the basis of per cent attainment in the fifth column of the

TABLE V.—TRANSFORMATION RATE IN THE FIRST STAGE OF THE WELD-QUENCH TEST AT 530 C.

All samples heated to 1350 C. for 1½ min. and held in first quenching bath, at 530 C. for time specified below, then water quenched. Hardness determinations were used to follow the degree of transformation before water-quenching.

Item	Hardness (Vickers-Brinell)			
	4 sec. at 530 C. and Water Quenched (A_{16} samples)	12 sec. at 530 C. and Water Quenched (A_{17} samples)	18 sec. at 530 C. and Water Quenched (A_{18} samples)	Standard Weld-Quench Test
2.....	397	248	243	224
3.....	534	291	287	260
17.....	459	267	219	221
18.....	481	321	298	246
19.....	487	385	359	267
22.....	493	492	470	377
24.....	588	584	591	450

TABLE VI.—MAXIMUM HARDNESS DATA FOR SINGLE-BEAD WELDS ON PREHEATED PLATES.

Item	Without Preheat (see Table II)	300 F. (150 C.) Preheat	500 F. (260 C.) Preheat	Hardness Change from No-Preheat to 500 F. (260 C.) Preheat Vickers-Brinell Units	700 F. (370 C.) Preheat
3....	266	254	227	39	212
9....	224	206	193	31	174
10....	287	....	206	81	200
11....	312	243	233	79	224
21....	243	206	193	50	195
22....	312	254	237	75	240
25....	345	250	224	121	224
26....	413	266	243	170	224
24....	503	....	262	241	....
28....	405	278	243	162	233
31....	397	243	237	160	233
33....	437	413	429	8	421
34....	383	363	390	+ 7	369
35....	383	270	230	153	237

maximum hardness values of the fourth column. The sixth column is therefore an indication of the degree to which the weld-quench test reproduced the area adjacent to the weld, on the basis of hardness alone. Micrographs of the weld-quench samples and of the area next to the weld which it was our aim to duplicate are shown in Figs. 5, 6 and 7 as additional evidence that the weld-quench test actually reproduced this area.

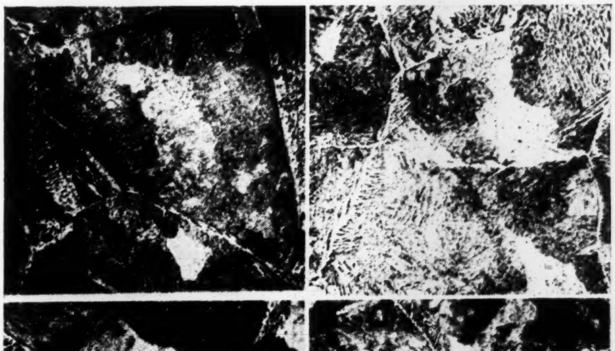


FIG. 5.—Comparison of Microstructure Adjacent to Weld with Weld-Quench Microstructure (× 250, reduced approximately one-half in reproduction).

In Fig. 8 the effect of alloying elements on the weld-quench impact value and the percentage loss of impact due to the treatment is shown graphically. It will be noted that the trend of these curves is downward with increasing carbon or alloy content—thus following the trend of welding experience.

Thermal Cycle During Welding:

The curves in Fig. 9 were obtained from the data provided by the motion

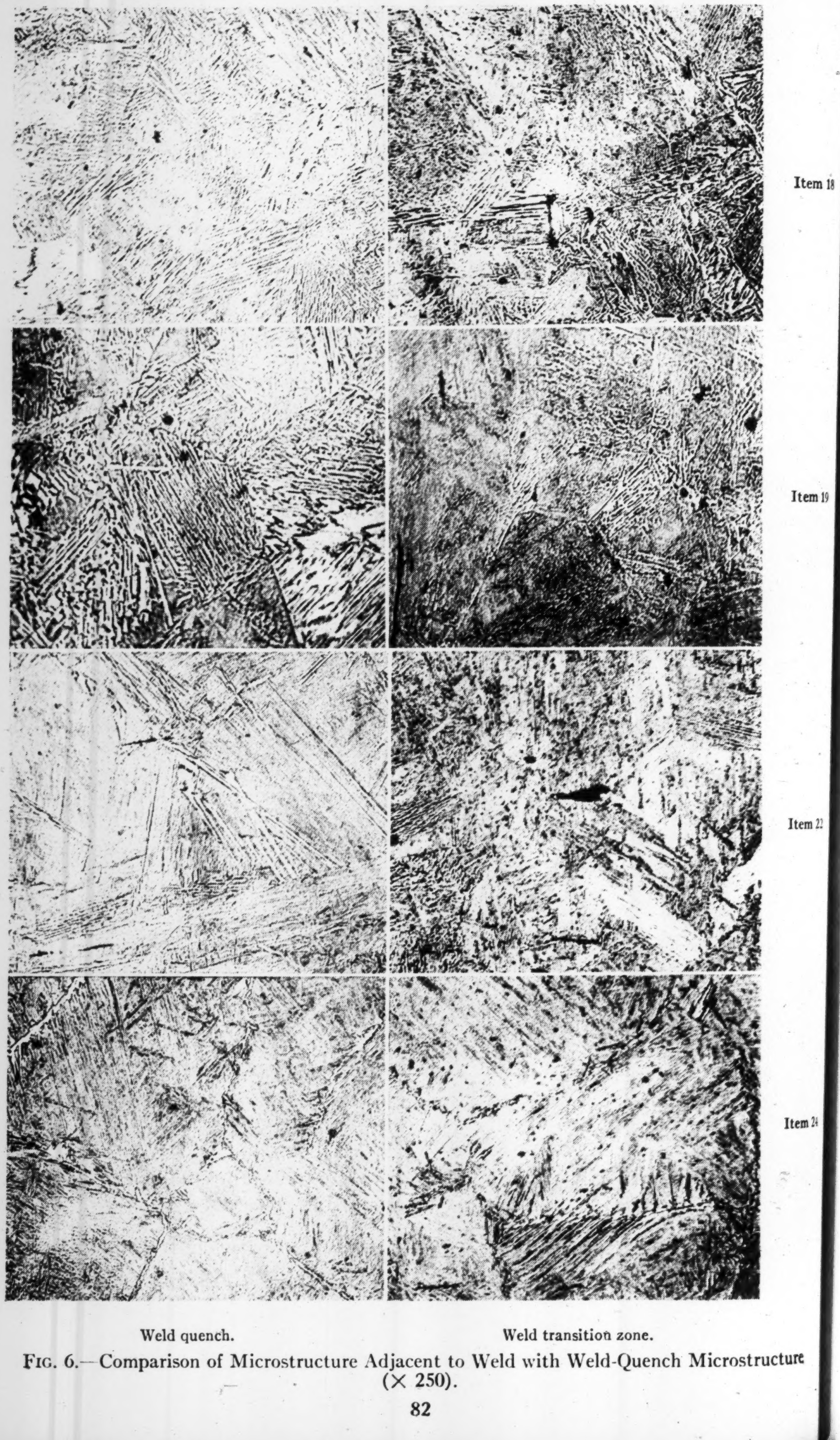
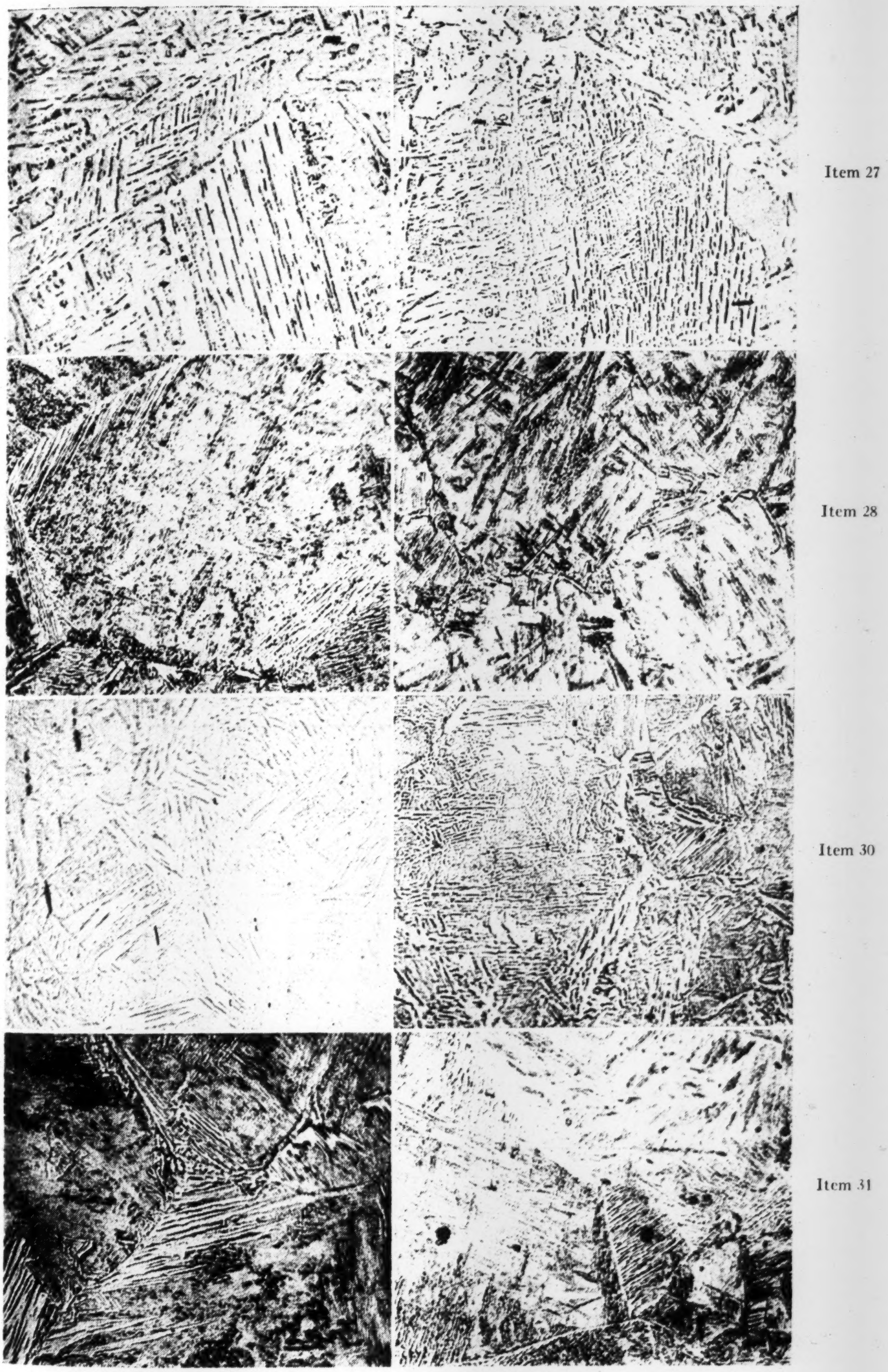

Item 18
Item 19
Item 22
Item 24
Weld quench.
Weld transition zone.

FIG. 6.—Comparison of Microstructure Adjacent to Weld with Weld-Quench Microstructure (× 250).


Item 27
Item 28
Item 30
Item 31

Weld quench.

Weld transition zone.

FIG. 7.—Comparison of Microstructure Adjacent to Weld with Weld-Quench Microstructure (× 250).

picture film. Curve 22W5 was obtained for a ½ in. thick, 3 by 6-in. plate of item 22 with the thermocouple bead approximately 0.02 in. from the fusion line. Curve 22W6 was for the bead 0.01 in., approximately, from the fusion line. Curve 4W5 was obtained for item 4 steel plate which was less than ½ in. thick. The welded plate was not sectioned after welding to determine the distance from the fusion line. Curves 22W5 and 22W6 were used to establish the time the weld-quench samples remained in the quenching baths.

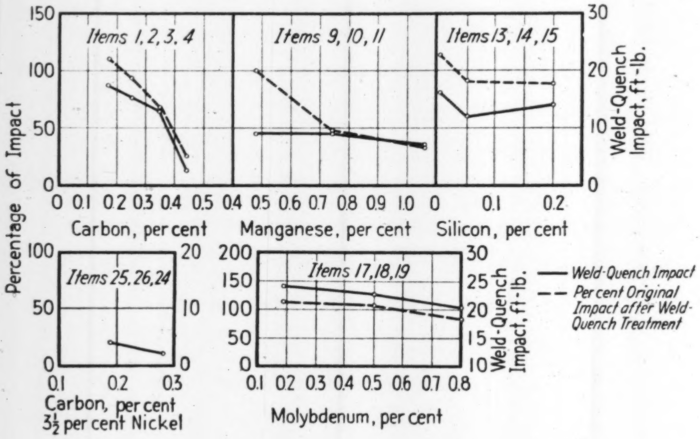


FIG. 8.—Comparison of Weld-Quench Values with Respect to Steel Composition

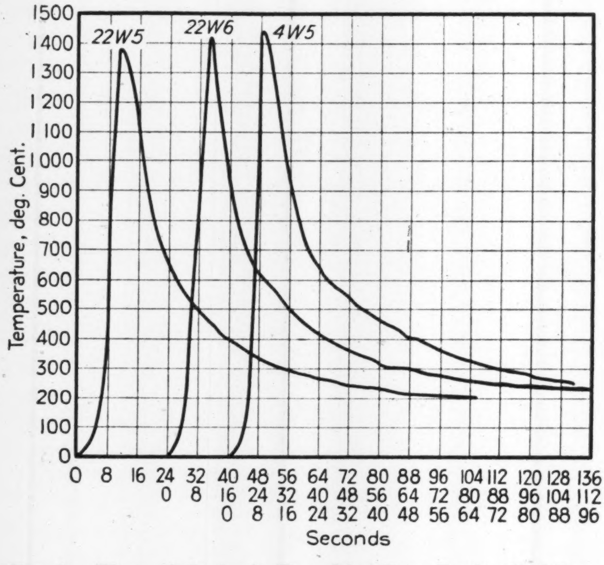


FIG. 9.—Thermal Cycles in Transition Zone During Welding.

A survey of the comparative rates of transformation for various steels with increasing carbon and alloy contents was made for the first quenching bath at 530

C. (see Table V). Figure 10 gives this survey in terms of hardness values reported on the various half-size Charpy samples quenched for the number of seconds at 530 C. as shown, then water quenched. A further comparison is made in the same figure with the weld-quench values for the same steels obtained in the standard two-stage quench at 530 and 310 C.

Finally, the hardness surveys on the single-bead welds made with preheated plates are given in Table VI and Figs. 11, 12 and 13.

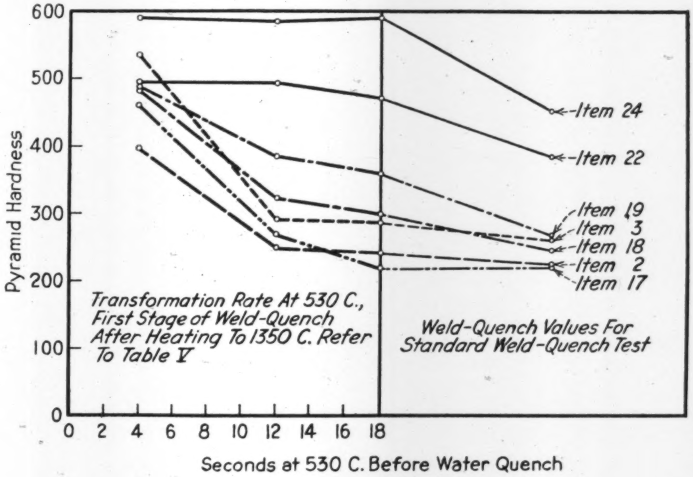


FIG. 10.—Transformation Rate of Weld-Quench Samples at 530 C.

DISCUSSION OF DATA

In the tabulated data for the maximum hardness found in progressive hardness surveys across the area adjacent to single-bead welds we find the facts to be the same as reported in the literature, namely, (*a*) carbon and alloying elements increase the hardenability, the carbon being the worst offender in this respect; (*b*) to increase the strength of the welded joint and at the same time keep hardenability low, we must increase the alloy content and decrease carbon to a low limit. There is always a question when hardness values alone are obtained for the weld cross-section as to what is too high an increase in hardness. Shall we limit hardness to an increase of 100 Brinell units, or stop just before we reach a martensitic structure? What is really sought is a quantitative statement of the inherent service hazard in using a joint with a microstructure corresponding to a certain hardness either left in the joint next to the last bead or produced during the welding operation and obliterated by subsequent welding beads or subsequent heat treatment. After the weld is made the questions to be answered are: (1) has the hardened zone adjacent to the weld cracked before the next bead was placed? (2) if no cracks have occurred will the hardened zone adjacent to the deposit resist dynamic and impact stresses without initiating cracks in this area? When we measure toughness of the parent material before it is welded and can determine what the toughness is after welding, a sensitive gage of the metallurgical damage is made available.

Considering the relationship of hard-

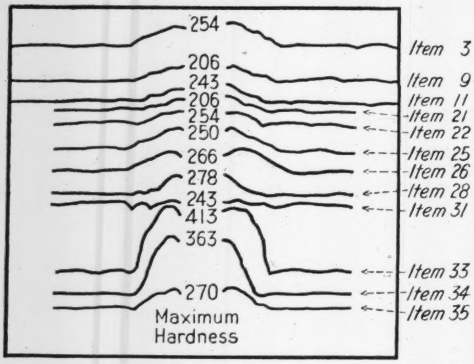


FIG. 11.—Hardness Contours for 300 F. (150 C.) Preheat.

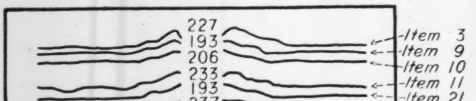


FIG. 12.—Hardness Contours for 500 F. (260 C.) Preheat.

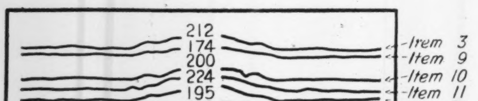


FIG. 13.—Hardness Contours for 700 F. (370 C.) Preheat.

ening in the heat-affected area of the single-bead welds of Table II and Fig. 2 to the toughness we find associated with this hardness by means of the weld-quench test, Table III and Fig. 8, we first note for the carbon steels that items 1, 2, 3, and 4 with increasing carbon show a gradually increasing hardenability. However, the weld-quench toughness value shows a marked drop beyond 0.35 per cent carbon—just where welding experience has placed the limit of carbon for good weldability of the plain carbon steels.

A comparison of items 27, 28, 30, and 31 for effect of carbon shows that the hardness increase for the first pair due to carbon is fivefold while the drop in weld-quench toughness is tenfold; for items 30 and 31 the hardness increase is threefold while the weld-quench value is reduced to one-fifth. Items 27 and 30 are two of many steels developed by the industry for easy weldability, and it will be noted that hardenability is very slight. The surprising data—incidentally quite reassuring for the welding industry—are the figures of 100 and 125 per cent of original impact value retained in the zone of items 27 and 30, respectively, where the drastic heating and quench of the welding operation has occurred.

The molybdenum series also deserves considerable attention inasmuch as increasing molybdenum does not contribute markedly to hardenability but does tend to lower appreciably the sensitivity toward the welding quench. For example, a mild steel, item 2, with approximately the same carbon as the molybdenum series items 17, 18 and 19 is lower in weld-quench toughness than item 19, the latter with considerably greater hardenability. Item 17 has practically the same hardenability as the mild steel, yet it shows an actual improvement in impact value for the weld-quench (incidentally a value which is almost twice as high as for the mild steel) over that for the original parent metal. In fact, item 17 with the lowest molybdenum content appears to be optimum for the series (items 17, 18, 19) with respect to hardenability and toughness adjacent to the weld bead, and it also compares favorably in every respect with the more complex steels of items 27 and 30.

The $3\frac{1}{2}$ per cent nickel series with carbon decreasing from 0.28 per cent to 0.19 per cent in the order of items 24, 26, and 25 presents another interesting case where a considerable reduction in hardenability is experienced with decreasing carbon in single-bead welds. On the assumption that hardenability is directly related to weldability, it would follow that we had improved weldability to a large extent. The weld-quench tests fail to bear this out since the values are quite close together and at an extremely low level, which confirms the industrial welding experience with these steels regarding their sensitivity to the welding quench. The welding methods required to bring out their excellent properties by preheating are discussed later in the paper.

With respect to preheating it may be noted that in Table II items 24, 28, 31 and 34 are ordinarily not considered as weldable without special handling such as preheating and possibly subsequent heat treatment as well. We would, therefore, not encounter in practice the high hardenabilities shown here for comparative purposes only. The above note should, therefore, be borne in mind in the discussion of comparative values of hardenability and weld-quench toughness.

The results on items Nos. 33 and 34 are such a case for we find that the addition of $\frac{1}{2}$ per cent columbium to the chromium-molybdenum steel definitely reduces hardenability, yet the weld-quench toughness of item No. 33 is high enough to compare favorably with a 0.25 per cent carbon mild steel while the addition of columbium has reduced the weld-quench toughness by one-half. Another observation here is that in view of the comparatively (compared with mild steel) high residual toughness of the straight chromium-molybdenum weld-quench test, in spite of a 61 per cent loss of original toughness, it does not appear surprising that cracks are reported to occur in the weld deposit, transverse to the weld, rather than in the hardened zone adjacent to the weld in this material.

A choice of best welding steels for easy weldability without preheating and on the basis of high parent metal hardness, low hardenability, high toughness before and after weld quenching (Table III) would include items 17, 18, 23, 27, 30 and 32. There are, of course, other analyses not included in the investigation that may show an equally desirable combination of properties, and it is recommended that the industry make available to the technical literature the weld-quench values of such materials. The six steels above are practically immune to the drastic heating and cooling adjacent to the weld deposit, they are not damaged metallurgically, and therefore, we are led to seek the reason. Part of the reason is low carbon, but the author believes that through his observation of a great many microstructures of welds and weld-quench specimens part of the answer is also in the ability of the steel in the heat-affected area to transform rapidly while still above 500 C., and so *uniformly* reject an alloy strengthened ferrite with a fine *uniformly distributed* dispersion of whatever carbide may be precipitated.

The data of Table V on transformation rates in the first quenching bath at 530 C. indicate the differentiation of easily weldable steels from those more sensitive to the thermal cycle. Figure

10 brings this out still more clearly; for example, it will be seen that items 2, 17 and 3 have practically completed their transformation in less than 18 sec., while items 24 and 22 show a considerable delay in transformation. The effect of increasing carbon and molybdenum in delaying the transformation may also be noted from the curves. The relationship of the weld-quench test to these curves and to the welding thermal cycle is an important consideration which can be followed by noting that a steel which is slow in transforming will not be completely transformed in the first quenching bath at 530 C. nor in the 18 sec. after the peak of the welding thermal cycle is reached. The lower part of the welding thermal cycle curve and the second quenching bath at 310 C. will, therefore, determine the properties of such steels, whereas the steels which transform rapidly at 530 C. are not at all sensitive to the second quench. The latter have already transformed completely and consequently their properties are set by the higher temperature transformation.

It must be noted here that a sensitive steel with considerable hardenability does not necessarily transform completely at 310 C. for there may be two modes of transformation of these steels as judged by the correlation of the single-bead welding tests and the weld-quench tests. The steel may have a delayed transformation and transform rapidly when some temperature between 530 and 310 C. is reached, or it may transform continuously at various rates (following the Bain S curve) down to 310 C. or lower. It is for this reason that the author advocates restricting the use of the weld-quench test to the following:

1. Determination of a suitable composition of the steel for foolproof welding.
2. Determination of an unsuitable composition for easy weldability with the further condition that such steels will require preheating and possibly subsequent heat treatment for successful welding.

The weld-quench test as far as it has been developed does not permit a classification to be made of the more difficultly welded steels. This would be a worthwhile project for future research as shown by the investigation of the effect of preheating, which has not been completed to the point of specifying the required degree of preheating by means of modified weld-quench tests.

A review of the data on preheating indicates in general the progressive benefit in lower hardenability for increasing preheat treatment. The larger drop in hardness for 300 F. (150 C.) preheat for the sensitive steels than for the others of lower hardenability suggests that most of the sensitivity is removed by preheating to this temperature and still more at 500 F. (260 C.) preheat. It is the author's belief that by a study of the single-bead welds on preheated plates and the preheat thermal cycle, weld-quench tests could be developed for determining the required degree of preheat and thus lead to classification of ease of weldability of the more difficultly welded steels by means of preheat temperature.

The lack of response to preheating experienced for items 33 and 34 is corroborating evidence of the difficult weldability of the steels as shown in the industry. The industry has found it necessary to preheat and reheat these steels after welding for complete annealing with the further precaution of not permitting the temperature to drop below 500 F. (260 C.) before reheating in a furnace. This procedure is a special treatment; therefore, the single-bead weld tests and the weld-quench tests based on them cannot be considered as reproducing this condition. For determining toughness adjacent to the weld for this special welding procedure it will be necessary to reproduce in a modified weld-quench sample the identical heat treatment for the entire procedure.

SUMMARY AND CONCLUSION

While the heat effect of the arc in welding mild steels was known in the past to produce grain growth, hardening and other effects which were considered to be non-critical, it is only comparatively recently that attention has been focussed on the heat effect due to the desire of the industry to provide structures with higher strength steels and constructed with the same remarkable ease as attended the welding of the mild steels.

The data presented on some of these mild, plain carbon steels justifies the arc welding industry's experience in considering the area adjacent to the weld of such steels as non-critical. A mild, steel with below 0.25 per cent carbon is shown to have practically the same toughness in the zone of largest grain and maximum hardness as before welding. When, however, the strength of such steels is increased by adding carbon, a serious reduction in toughness of the weld quench results. Where higher strength is obtained by addition of alloying elements the hardening effect of the added element is additive to that of carbon and may intensify it. This intensifying effect is not the same, quantitatively, for all the alloys. An example is provided in the molybdenum steels, items 17, 18 and 19, where the alloy has improved the weld-quench toughness over that of a mild steel with no decrease in carbon. The steel mills have taken cognizance of the need for steels of higher strength which will not develop high hardness and give low toughness next to the weld bead. They have produced steels of low alloy contents which have the equivalent strength of higher carbon steels without the welding hazard associated with the latter. Items 23, 32, 27, 30 are such low-alloy steels and the reason for their successful application in the welding field is shown by the high weld-quench values. The steels are tough after going through the drastic heating and cooling next to the weld; therefore, the region adjacent to the weld is not a critical one in such a welded joint.

The weld-quench test developed in the Naval laboratories is one which can be applied to the parent steel before it is welded, even before it is rolled to plate size for welding, and should be attractive for the steel mills and consumers of steel for structural welding. The test can be made at a low cost and will indicate whether the composition of the steel is suitable for easy welding or whether preheating will be necessary. It will make possible the matching of heats of steel with steel already in successful welding use in the consumers' plant. The test should be a useful one for the consumer for putting new steels in their proper niche as to ease of weldability.

The essence of the weld-quench test is that it separates steels for welding into two groups: those insensitive and those sensitive to the arc welding thermal cycle. The insensitive steels are those that transorm to soft, tough products at a rapid rate, and at a high temperature. The sensitive steels have a delayed transformation and the latter takes place when the rapidly decreasing temperature of the weld heat has dropped to a low level. Hard, brittle products are the result of this low-temperature transformation.

Where sensitive steels were encountered a low weld-quench toughness was obtained. With preheat temperature in single-bead welding, these same steels gave evidence of decreasing hardenability with increase in preheat temperature. It is to be expected that, with the softer microstructure obtained by preheating, the toughness should be increased. However, the increase in toughness was not determined since modified weld-quench tests have not yet been developed to reproduce the preheat condition.

We can, therefore, conclude that the weld-quench test in its present state of development can select good welding steels of easy weldability. Furthermore, it shows that when a suitable composition of steel is chosen the critical area adjacent to the weld is not deficient in desirable properties and has a low hardenability. Further extension of the weld-quench method to the problem of the weldability of the more sensitive steels may lead to the solution by quantitatively classifying their weldability on the basis of the preheat required to give high toughness adjacent to the weld. The remaining problems of the maximum plate cross-section for easy weldability before preheating is necessary and the maximum speed of welding without appreciable reduction in toughness may also be put on a quantitatively determinable basis by further study of the thermal treatment of the area in question.

Acknowledgment:

The helpful counsel of R. H. Canfield, Superintendent of Division of Metallurgy, Naval Research Laboratory and H. W. Hiemke, Welding Engineer, Specification Division, Bureau of Engineering, Navy Department is gratefully acknowledged. The author also wishes to thank the Director of the Naval Research Laboratory and the Chief of the Bureau of Engineering for permission to publish the results of the investigation.

The aid of H. F. Taylor of the Naval Research Laboratory Staff in preparation of materials and heat treatment is also acknowledged.

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DISCUSSION

MR. HUGO H. HIEMKE[1] *(presented in written form).*—A simple and reliable test for weldability of steel is something that the welding industry has looked forward to for some time. The Industrial Research Division of the Engineering Foundation Welding Research Committee is now considering a program of research which has as its objective the development of just such a test for weldability. The timeliness of Mr. Bruckner's contribution may therefore be more fully appreciated.

Naturally, in this Symposium on Impact Testing, the question of fundamental interest in all of the papers presented is the correlation between the values recorded in the impact, or notched-bar, test, and the suitability of the metal being tested for some particular service. More specifically, in the present instance, we are interested in the correlation between the weld-quench impact value and the weldability of steel.

There has been some criticism of the use of the Charpy test for evaluating weldability on the grounds that this test is too controversial; it is too difficult to reproduce results, and interpret these results after they are recorded. Others have criticized the testing of a separately heat-treated bar, on the grounds that this cannot represent the thermally-affected zone of a weld as well as an impact test taken transverse to the welded joint. The following comments are offered in answer to these criticisms, and in defense of the procedure developed by Mr. Bruckner.

The term "weldability," as used in this discussion will be defined as "the ability of a steel to be subjected to the thermal effect of an electric arc weld without appreciable hardening or loss of toughness." Stated in a slightly different manner, we want steels for welding which will not crack during welding, and whose properties will not be so adversely affected by the welding operation that they are rendered unsuitable to resist service stresses.

During the welding process, the metal adjacent to the weld is subjected to two kinds of thermal effects—metallurgical and mechanical. The metallurgical effects include changes in properties and structure, which have been discussed at some length in the author's paper. The mechanical effect is the production of thermal stress, due to the unequal temperatures of the weld metal and the base metal. These stresses are biaxial, and are of sufficient magnitude to exceed the yield point of the base metal and the weld metal. In order to appreciate the complete thermal effect we must visualize the grain growth, the hardening, and the plastic yielding going on simultaneously. To evaluate weldability, we must look for a test which measures the toughness of the steel, or the ability of the steel to deform plastically under biaxial stress. In the opinion of the writer, the notched-bar test most satisfactorily fulfills this requirement. Without going into the theory of the notched-bar test in detail, suffice it to say that the excellent correlation between the weld-quench impact values and the field experience in the welding of these steels tends to confirm this opinion. Those steels which have been found difficult to weld under shop and field conditions without preheating or postheating, produce low weld-quench values while those steels which have been welded satisfactorily in shop and field produce high weld-quench values.

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In regard to the testing of impact specimens transverse to welds to determine the weldability of steel, the following comments are offered. This procedure necessarily requires the production of a test weld, of sufficient thickness to yield a Charpy bar. The welding technique, which includes the type and size of electrode, the amperage, voltage, welding speed, the preheat, interpass, and postheat temperatures, and the beading sequence, exerts an influence on the properties of the thermally affected zone of the steel. Such welds would ordinarily be prepared by hand-welding, introducing another variable. In addition to these variables of technique, there is the very serious problem of locating the notch of the impact specimen exactly at the critical zone which is being studied. Since the thermally-affected zone is narrow and irregular in shape, the notched section is likely to be made up of a variety of structures, rather than the highly overheated structure whose properties are significant in the study of weldability. The writer has no quarrel with the use of the notched-bar test of transverse (and longitudinal) weld specimens as a proof test of welded pressure vessels, but as a method of evaluating the differences in weldability between two steels, this method leaves much to be desired.

TABLE I.—IMPACT AND HARDNESS DETERMINATIONS IN WELD-QUENCH TESTS ON 1/2 IN. ROLLED STEELS.

Designation	Chemical Composition, per cent						Impact			Vickers Hardness		
	Carbon	Manganese	Molybdenum[a]	Nickel[a]	Copper[a]	Phosphorus[a]	As Received, ft-lb.	Weld-Quench, ft-lb.	Ratio, Weld-Quench to As Received, per cent	As Received	Weld-Quench Samples	Ratio, Weld-Quench to As Received, per cent
Carbon steel	0.26	0.67	......	......	......	......	19	19	100	165	236	144
Carbon steel	0.20	0.70	......	......	......	......	15.5	16	103	168	212	126
Mn-V steel	0.18	1.18	V.07	......	......	......	15.5	16	103	208	234	112
C-Mo steel	0.08	0.76	0.44	......	......	......	28	27	96	149	217	145
0.10 C-Ni-Cu steel	0.09	0.42	......	1.94	1.00	......	23	25	109	163	208	128
0.14 C-Ni-Cu steel	0.14	0.57	......	1.99	1.02	......	20	21	105	177	212	125
Cu-Ni-Mo-P steel	0.09	0.75	0.10	0.72	1.63	0.097	11.5	16	139	202	234	115
Cu-Ni-P steel	0.09	0.56	......	0.59	1.04	0.112	13	14	108	169	206	121
Ni-Mo steel	0.16	0.59	0.10	1.90	......	......	14	16	114	165	219	133

[a] Nickel, copper, molybdenum, and phosphorus analyses recorded only for steels having more than usual residual content. All steels were silicon-killed, containing 0.15 to 0.25 per cent silicon, except the Cu-Ni-Mo-P alloy, which contained 0.06 per cent silicon.

The weld-quench test developed by Mr. Bruckner establishes the property of the worst structure encountered adjacent to a single-bead weld made with $\frac{3}{16}$-in. covered electrodes on $\frac{1}{2}$-in. steel plate at 180 amp., 27 v., and 6 in. per min. forward travel, without preheating or postheating. These conditions, while arbitrarily selected, represent something definite and reproducible. The weld-quench values are determined on steel which has been subjected to a definitely controlled thermal cycle, dependent only on the control of time and temperature. This procedure is therefore viewed as a significant forward step in the evaluation of the weldability of steel.

Since Mr. Bruckner left the Naval Research Laboratory, another series of steels has been investigated, using the weld-quench procedure. These steels are all from commercial heats, $\frac{1}{2}$ in. in thickness, and are all produced primarily as fool-proof weldable grades of steel. That is, they are intended for welding in locations where preheating and postheating are not practicable. The chemical analyses, and impact and hardness values are tabulated in the accompanying Table I.

It is significant that all of these steels produced weld-quench values well in excess of 10 ft-lb., and that the ratio of the weld-quench impact value to the "as-received" impact value was in all cases over 96 per cent. The hardness numbers of the weld-quench specimens were in no case in excess of 150 per cent of the hardness of the steel in the as-received condition. These steels would therefore be considered weldable, by definition, since they may be "subjected to the thermal effect of an electric-arc weld without appreciable hardening or loss of toughness."

MR. A. B. WILDER[2] (*presented in written form*).—The weld-quench test as described in this paper should be of interest to the welding industry and manufacturers of steel. It would be worth-while for industry to utilize this test and acquire further information regarding its adaptability to the solution of a fundamental problem.

The heat treatment specified for the test appears to duplicate, in general, conditions in the heat-affected zone. It would appear that the hardness and microstructure in certain steels could not be duplicated by one standard method of subcritical quenching, due to marked differences in reaction rates. The weld-quench test depends upon reaction rates and because of this fact the question arises as to whether the treatment at 310 C. for 24 sec. could be eliminated. Quenching certain steels from 530 C. after a period of 18 sec. would produce a large amount of martensite which may help to increase the sensitivity of the test.

The author has indicated that hardness is not necessarily an indication of weldability—the term weldability being considered as a criterion of the properties of the heat-affected zone in a single weld bead. It should be pointed out that impact is not a universal criterion of weldability when applied to many types of welded structures. The short time consumed by making a single weld bead and determining the hardness of the heat-affected zone may offer further possibilities as a weldability test.

Considering the subject of intergranular corrosion of stainless steels, the weld-quench test would have to be modified to meet this condition. Materials of this type present a problem which is different from the one under general consideration. It would be of interest to have the opinion of Mr. Bruckner on this particular subject.

The preheating of alloy and high-carbon steels is an important consideration for the welding industry. The author has pointed out that this condition could be duplicated in a modified weld-quench test. It would be an important contribution to the science of welding if a simplified test could be devised to determine the amount of preheating necessary. This of course would necessitate a comprehensive program of research and it would appear that a modified weld-quench test may provide the necessary information.

MR. A. B. KINZEL.[3]—This paper presents an elegant study of the microstructures produced in welds as they are made in practice and a remarkable comparison with the structure of synthetic coupons. It teaches us much about what happens when a welded plate cools. It is very much worth-while, and I wish to congratulate the author. There are several comments that might be made on the author's suggestion that his method might be developed into a routine test. In effect the author takes three points on the *S* curve and assumes that any given steel is defined by those three points. That is not necessarily so, particularly for a new steel about which we know little. In this event it is necessary to correlate this test with a straight welding test involving subsequent Charpy impact or some other equivalent. If we are to perform such a test, why bother to go through with the weld quench later? An illustration is specimen 34, a chromium-columbium steel, showing 7 ft-lb. and condemned by the weld-quench test. Other tests made on such material taken from the weld, as well as extensive service, have shown that that material is entirely weldable from the standpoint of toughness next to the weld.

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[2] Assistant Professor of Metallurgical Engineering, University of Illinois, Urbana, Ill.

[3] Chief Metallurgist, Union Carbon and Carbide Research Laboratories, New York City.

The difficulty of checking coupons taken from a weld is not quite as great as is indicated by the previous discussion. By taking a Charpy bar slightly longer than usual, and giving it a very light etch, the drill can be spotted at the scarf or at any point adjacent to the scarf with very good accuracy. We are using this method right along in testing steels, and have made hundreds of such tests. We are able to check the service performance of the well-known steels as well as other qualities and properties of some of the newer steels with which we are working. Thus, while this paper is a valuable contribution and teaches as much, I certainly would be very much opposed to the test procedure being introduced as common practice to determine weldability. The method also fails to take into account mass effects. For example, chromium-molybdenum steel with 0.30 per cent carbon, in this and many other tests, will prove to be unweldable, yet we all know that with the technique developed in the aircraft industry, and the thin-walled tube common in the aircraft industry, this steel regularly is being and has been welded for the past 15 yr. without getting brittleness next to the weld.

It might also be well to reject the definition of welding which excludes all methods of welding except the electric arc! Other methods such as oxyacetylene welding or resistance welding set up very different temperature gradients, bring us to a different slice on the *S* curve and again demand an actual weld as a criterion. Thus, while we again wish to congratulate the author, we hope that he will not attempt to make a routine test out of his elegant metallurgical method.

MR. WALTER H. BRUCKNER[4] (*author's closure by letter*).—I wish to thank those who have contributed to the discussion, thereby extending the data of the paper and presenting an opportunity for the author to re-emphasize certain aspects of the steel weldability problem. The additional data which Mr. Hiemke has presented are satisfying in that the high weld-quench values obtained for the fool-proof welding steels corroborates the assertion made in the paper that properly chosen steels do not suffer metallurgical damage next to the weld bead, made with a d.c. heavily coated electrode. Mr. Hiemke's analysis of the weld-quench test and his comments on the correlation of the test values with those from shop and field welds are appreciated as adding strength to the author's conclusions.

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[4] Research Associate in Metallurgical Engineering, Engineering Experiment Station, University of Illinois, Urbana, Ill.

In answer to one of the questions raised by Mr. Wilder we need to go back to the starting point for the weld-quench test, that is, the ½ in. thick plates of the steels which were welded with a single bead. After we obtained the maximum hardness, grain size, and microstructure of the overheated area adjacent to the weld we prepared sections of the original parent metal (which has not been welded or heat treated) and gave this material the weld-quench test described in the paper. If there is any difference in the reaction rates of the steels this will be reflected in differing hardness and impact values respectively in the welded plate and weld-quench test specimens. If the weld-quench test did not approximate the thermal cycle adjacent to the single bead weld we would not find the close similarity of microstructure which was obtained. We are reminded also by the data that hardness values are closely reproduced; therefore since grain size, microstructure, hardness, and thermal cycle are the same we are reasonably sure that the weld-quench test bar is a physically magnified section of the overheated area adjacent to the weld. However, if we change the conditions of preparing the single bead welds from those described in the paper, the weld-quench test therein described cannot be regarded as matching the new conditions.

Mr. Wilder's second question is adequately answered in Mr. Hiemke's discussion regarding the correlation of weld-quench values with field and shop welding experience. With regard to the stainless steels the critical area of intergranular corrosion in such steels as are susceptible to this effect in welding occurs at a distance from the weld and is not due to overheating but rather reheating of austenite at temperatures between 1100 and 1600 F. (595 and 870 C.). The precipitation of grain boundary carbides from an austenite unstable in this range of temperature is an entirely different problem than that considered by the author. This problem has also been solved quite satisfactorily by the use of low-carbon material and the use of stabilizing alloys in the stainless steel plate and electrode.

It might be well at this time to state the conditions under which the weld-quench test applies:

1. For hypoeutectoid, structural, carbon, or low-alloy steels.
2. For steels as in (1) welded by means of the heavily coated, d.c. electrode.

Mr. Wilder's comments on the use of preheat are answered below in commenting on Mr. Kinzel's observations.

For the benefit of those who may infer from Mr. Kinzel's discussion and the author's data on certain steels showing low weld-quench values that the weld-quench test will be used as a means of condemnation of such steels for welding we are forced to reiterate certain views expressed in the paper.

The paper as presented is in the nature of a progress report in that the weld-quench test developed separates steels for welding into two categories:

1. Those easily welded.
2. Those not easily welded.

It is recognized that in the steels of group 2 are those having excellent properties required for special structural or service conditions. However, in order to obtain these high properties in the completed welded structure, it is necessary to exercise special care, preheat and occasionally preheat and postheat treatment during the welding of the structure. The weld-quench tests whereby the toughness of the steels in group 2 may be obtained for these special conditions of welding are not available as yet, but attention has been directed in the paper to the value of additional research to complete the original program.

The author is not interested in condemning any steel for welding use; rather he is interested in defining the conditions for successful welding. When a modified weld-quench test based on the procedures differing from easy weldability has been established there will be available a means of classifying the suitability of steels for welding in group 2.

In answer to the second point raised by Mr. Kinzel, regarding the restriction of the transformation of austenite to temperatures around 530 C. for steels with rapid rates of reaction and to 310 C. for sluggish steels in the weld-quench test, it would appear that for welding we should choose just such a restriction of high-temperature transformation either by choice of fool-proof steels or conditions of welding. We do not want the transformation to take place at the lower temperatures to form acicular brittle products. With steels in group 2 we must define successful weldability as those conditions of welding which produce a thermal cycle with sufficient time at the high temperature to permit the formation of pearlite.

The illustration given by Kinzel for thin-wall tubing, gas welded for aircraft use, is such a case, for the thin wall and gas torch provide a thermal cycle next to the weld bead which permits decomposition of austenite to tough ductile pearlite. The same steel in thicker section and welded with the d.c. heavily-coated electrode might be classed as a difficultly welded grade. The facts are as Kinzel has presented them, that is, a steel difficult to weld with the metallic arc may be weldable under the gas torch. However, the experience of the industry with the d.c. arc and heavily-coated electrode favors this welding method because of the ease and rapidity of fabrication together with the desirable properties obtained and minimum distortion.

With regard to the use of the actual weld joint for notch impact tests as described by Kinzel it is obvious that the volume of metal deformed in fracturing includes the region of high hardness adjacent to the weld, part of the weld metal and part of the overheated metal more remote from the weld bead. The machining of the notch in such a sample also may have removed the most critical area in the actual weld. The values reported by such a test are therefore never as low as a test of a bar of metal with exclusively the most critical microstructure of the actual welded joint. In actual dynamic service the weld joint made with unsuitable steel fails by cracking through the most critical region we have considered in the weld-quench test. This critical area covers only a microscopically small extent in the weld joint but a crack extending through the plate even though confined entirely to this region means complete failure of the structure.

There is another question related to the weld-quench test which has not been broached by the discussion and that is the translation of the properties determined by the weld-quench test to the actual weld joint. It is known that the weld joint is a microstructurally heterogenous gross structure and that continuity of stress across the joint demands as nearly homogeneous mechanical properties as possible. It has been stated in the paper that welding steels suitably chosen do not show metallurgical damage adjacent to the weld because of the high toughness that is retained in the overheated parent metal. It is however possible to have good toughness in this region and still have the joint fail by cracking in dynamic service because of the juxtaposition of low-strength weld metal which becomes a metallurgical notch. The weld-quench test is therefore of value not only for gaging the suitability of the steel used for welding but also for determining the properties which the weld metal must possess if the tough high-strength properties of the area adjacent to it are not to concentrate the stress in the weld metal and thus make for early failure.

The author looks forward to the extension of the use of the weld-quench test to other steels and other conditions of welding than those he has investigated. It is his belief that wider use of the test will extend the application of welding to steels which are at present regarded with suspicion. It will be noted that the Strauss test for testing the intergranular corrosion susceptibility of the welded stainless steels has done much to remove the hazard of welding and led to the solution of the problem by means of low-carbon and stabilizing alloys. It is the author's hope that the weld-quench test may do for the low-alloy structural steels what the Strauss test has done for the stainless grades.

STRESS-STRAIN RELATIONS UNDER TENSION IMPACT LOADING

By D. S. Clark[1] and G. Dätwyler[1]

Synopsis

This paper discusses a method of determining force-elongation diagrams for metals under tension impact loads. A new type of tension dynamometer is described, and results of tests on a few steels and non-ferrous metals and alloys at a velocity of about 11 ft. per sec. are given. The results indicate that for most of the materials tested the yield forces under dynamic conditions are higher than those under static conditions, and that, in general, the maximum dynamic forces are greater than the maximum static forces. It has been shown that in some cases energies under dynamic loading are greater than those given by the stress-strain curve as modified by Mann. Experimental evidence is given to show the reliability of the method developed.

The behavior of a material under conditions of tension impact loading can be shown to best advantage by means of a force-elongation diagram. To accomplish this, the authors have developed new equipment for determining a force-time curve in tension and a method of reducing this to a force-elongation diagram.

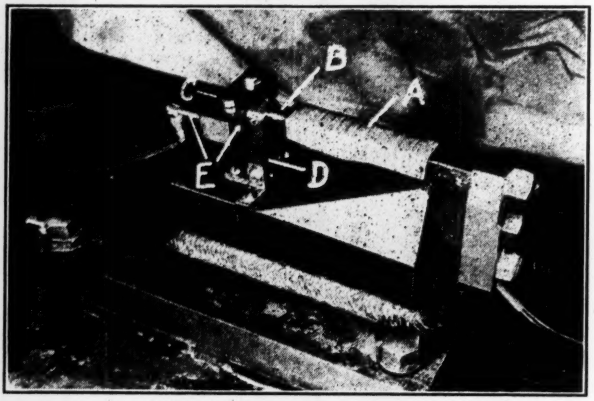

Fig. 1.—Dynamometer.

Apparatus

For the purpose of discussion, the main items of the equipment may be divided into two parts: first, the dynamometer for measuring the forces; and, second, the recording apparatus for correlating force and time.

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[1] Assistant Professor of Mechanical Engineering, and Research Fellow in Aeronautics, respectively, California Institute of Technology, Pasadena, Calif.

Dynamometer:

The dynamometer shown in Fig. 1 consists of a rectangular bar $\frac{7}{8}$ by $\frac{3}{4}$ in., with a uniform cross-section over a length of $4\frac{5}{8}$ in., machined from S.A.E. No. 1055 steel. At one end of the bar a $\frac{1}{2}$-in. hole is drilled and tapped for 20 threads per inch, into which one end of a specimen may be screwed. The other end of the dynamometer is bolted to an anvil mounted in a 120 ft-lb. Olsen Izod impact testing machine with a maximum velocity of 11.3 ft. per sec.

Approximately 14 ft. of No. 40 constantan wire is laid longitudinally on the four successive faces of the bar in "zig-zag" fashion and coated with glyptal as a binder. For purposes of protection, the wire is wound with scotch tape as shown at *A* in Fig. 1. This resistor is referred to as the "pick-up."

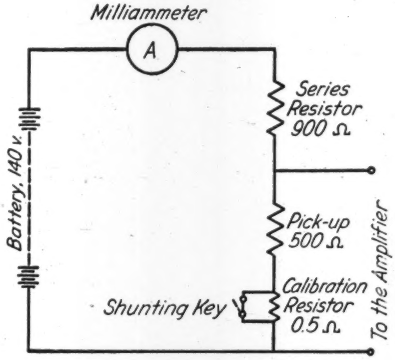

FIG. 2.—Pick-up Circuit.

The unnotched specimen *B*, 0.20 in. in diameter over a gage length of 1.0 in., is screwed into the end of the dynamometer against a stop to eliminate play in the threads. A tup, shown at *C* in Fig. 1, is screwed onto the other end of the specimen and clamped in proper position. The tup and specimen are held in alignment by the guide *D*.

The leads from the pick-up on the dynamometer are connected to the circuit shown in Fig. 2. This circuit consists of a 140-v. battery, a resistor (series resistor) placed in series with the pick-up, and a resistor for purposes of calibration as discussed later. The series resistor has such a value that a current of approximately 100 ma. is maintained in the circuit.

When the tension impact forces act on the specimen, they are transmitted through the specimen to the dynamometer, which elongates elastically in proportion to these forces according to Hooke's law. The wire covering the bar follows this increase in length, thus changing its resistance. Numerous tests, of which the data in Table I are typical, indicate that the change in resistance varies linearly with the load over a range exceeding that used in the tests reported. It will be noted that the resistance for loading and unloading agree within limits of experimental error, indicating that the Glyptal binder is sufficiently strong to permit the wire of the pick-up to follow the change in length of the dynamometer under load. By measuring the change in voltage produced by the change in resistance, the forces can be determined as indicated later.

TABLE I.—TYPICAL CALIBRATION OF RESISTANCE *versus* LOAD FOR DYNAMOMETER.

LOAD, LB.	RESISTANCE, OHMS
0	457.890
4 000	458.080
8 000	458.269
12 000	458.450
8 000	458.262
4 000	458.079
0	457.889

The linear relation between the impact forces and the dynamometer deformations is valid as long as the period of the natural vibration of the dynamometer bar is short compared with the duration of impact. The natural frequency of the dynamometer developed in this work is 16,000 cycles per second, which gives a time for a half period of 1/32,000 sec. The natural frequency was determined by striking the end of the dynamometer and by comparing the vibrations, set up on an oscillograph screen, with a wave of known frequency. The duration of impact is of the order of 2/1000 sec., as shown by the test oscillograms discussed later.

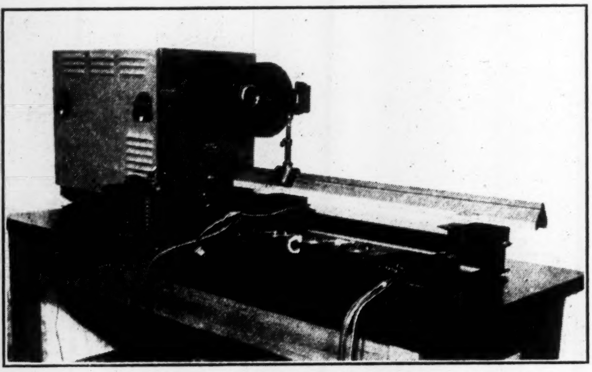

FIG. 3.—Recording Apparatus.

Recorder:

The voltage changes in the circuit, produced by the elastic deformation of the dynamometer, are amplified by a one-stage alternating current amplifier. From the amplifier the signal is sent to an R.C.A. TM-168-A cathode ray oscillograph, shown at A in Fig. 3. The connections are made such that the voltage changes will produce a vertical deflection of the electron beam. The scale for the vertical deflection in terms of resistance (ohms per inch) is determined by suddenly changing the resistance of the dynamometer circuit a known amount. For this purpose, the calibration resistor indicated in Fig. 2 is used. The change in resistance of the dynamometer winding for given loads may be determined statically in a tension testing machine, as shown in Table I.

The circuit of the oscillograph is arranged so that a single horizontal sweep of the electron beam can be obtained at any desired speed. The sweep speed is determined by connecting a known frequency to the oscillograph.

The force-time curve traced by the beam on the screen of the oscillograph is recorded photographically with a camera shown at B in Fig. 3. The camera is equipped with a Zeiss Biotar f:1.4 lens.

The horizontal sweep of the oscillograph is started slightly in advance of the impact by extending the pin shown at E in Fig. 1 a short distance ahead of the tup. This pin is insulated from the tup and connected to a condenser in the control panel shown at C in Fig. 3. The other side of the condenser is connected to ground. When the hammer of the pendulum strikes the end of the pin, the condenser is discharged, thereby starting the sweep of the electron beam. An instant later, the hammer strikes the tup, producing a force-time diagram in the center of the oscillograph screen.

FORCE-ELONGATION DIAGRAM

Since the records taken on the oscillograph give force-time curves, it is necessary to convert them to force-elongation curves. This conversion is made in the following way:

TABLE II.—AVERAGE RESULTS FROM STATIC AND DYNAMIC TENSION TESTS.

Material	Yield Point, lb.		Maximum Load, lb.		Elongation in 1 in., per cent			Reduction of Area, per cent		Energy			
					Measured		Calculated			Static		Dynamic	
	Static	Dynamic	Static	Dynamic	Static	Dynamic	Dynamic	Static	Dynamic	Engineering Curve	Potential Curve	Pendulum	Curve
S.A.E. No. 6140[a]	....	....	9667	9873	4.4	9.7	12.8	11.6	34.1	29.0	30.9	88.5	85.3
S.A.E. No. 1015[b]	2650	3521	2803	3607	11.7	16.6	16.1	48.1	50.7	25.3	31.3	43.1	42.6
S.A.E. No. 1018	1175	2343	1803	2518	35.7	35.4	34.0	70.6	66.8	48.5	67.4	65.0	64.8
18-8 Alloy	3033	3713	3847	4665	39.0	26.6	26.9	69.0	53.9	116.2	164.2	98.6	103.7
Duralumin 17S-T	1100	1423	1673	1832	22.0	22.0	21.3	44.9	46.5	28.3	33.8	29.9	31.7
Brass[b]	1383	1918	1772	2024	22.0	25.6	28.3	44.4	50.5	31.6	38.3	46.1	46.6
Aluminum	500	761	594	786	15.1	24.6	26.6	69.2	75.7	6.51	9.22	15.0	14.9
Copper	1200	1720	1327	1845	12.7	19.4	19.2	58.0	57.2	12.6	16.7	26.7	26.8

[a] Oil quenched from 1620 F.
[b] Cold-rolled.

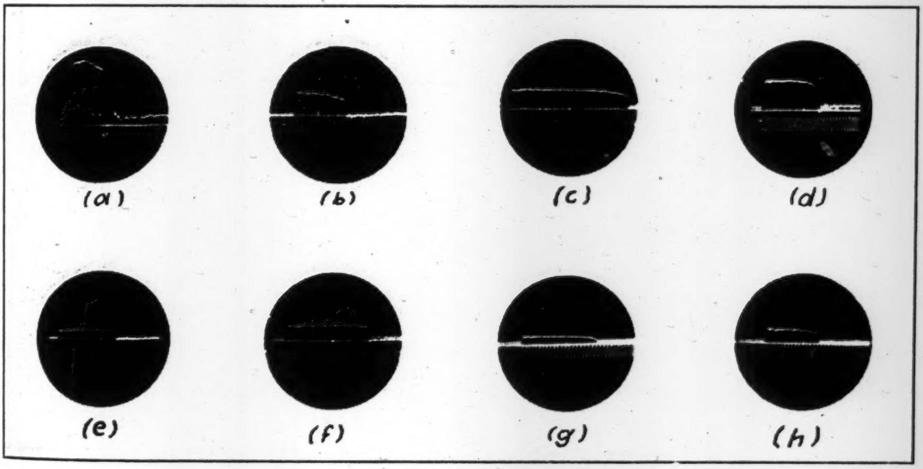

FIG. 4.—Representative Force-Time Diagrams.

(a) S.A.E. No 6140. (b) S.A.E. No. 1015. (c) S.A.E. No. 1018. (d) 18 per cent chromium, 8 per cent nickel. (e) 17S-T duralumin. (f) Brass. (g) Aluminum. (h) Copper.

The force-scale and the time-scale factors for the diagram under consideration are determined. The diagram is then photographically enlarged and traced by drawing a mean line through the vibrations in the diagram. The intersection of this mean line with the ascending line at the beginning of the diagram is defined as the "dynamic yield point." The tracing is then divided into convenient time intervals. The area of each of the interval sections is determined by multiplying the average force over this interval by the time interval. By means of the calibration, the areas are given in units of momentum. These values of momentum are

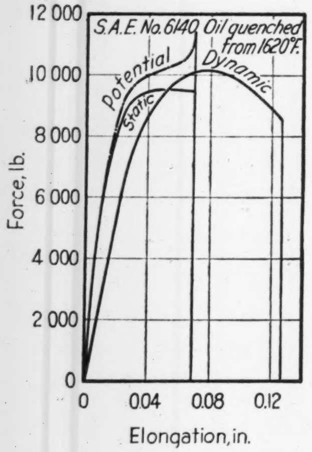

FIG. 5.—Force-Elongation Diagram, S.A.E. No. 6140.

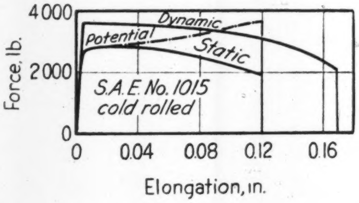

FIG. 6.—Force-Elongation Diagram, S.A.E. No. 1015.

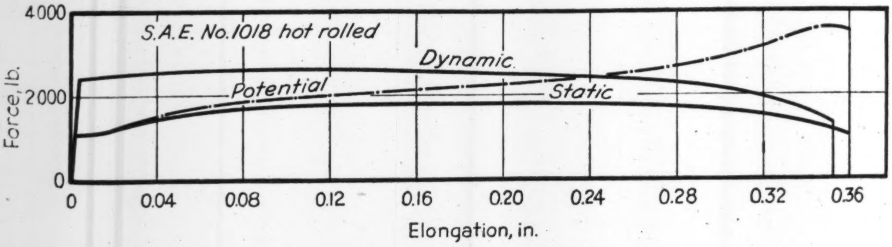

FIG. 7.—Force-Elongation Diagram, S.A.E. No. 1018.

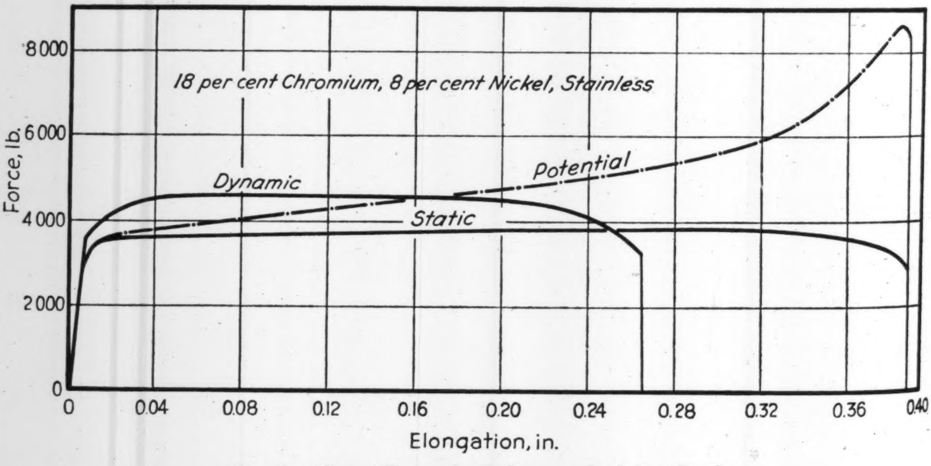

FIG. 8.—Force-Elongation Diagram, Stainless Steel.

divided by the mass of the pendulum, thereby giving the decrease in the velocity of the pendulum. By subtracting these values from the initial velocity of the pendulum, the relation of velocity-time is obtained. Using the same time intervals, the average pendulum velocities are multiplied by the time interval, thus giving the change in elongation for each interval. These individual elongations are added cumulatively to give the total elongation at any point. From the force-time and the elongation-time relations, the force-elongation curve is obtained. The final elongation from this curve is referred to as the "computed elongation."

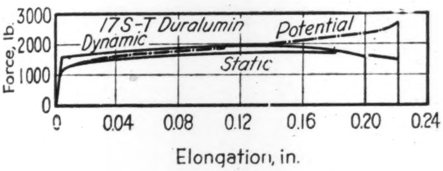

FIG. 9.—Force-Elongation Diagram, Duralumin.

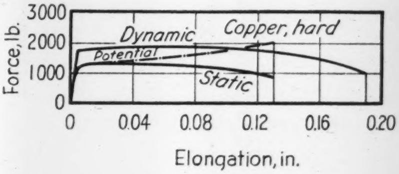

FIG. 12.—Force-Elongation Diagram, Copper.

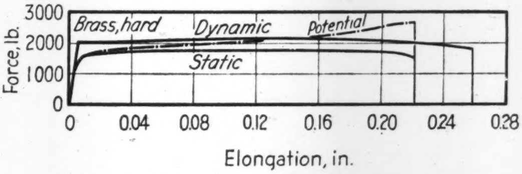

FIG. 10.—Force-Elongation Diagram, **Brass.**

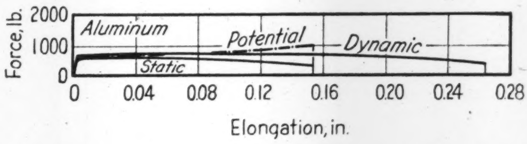

FIG. 11.—Force-Elongation Diagram, Aluminum.

It is assumed that the rate of strain is equal to the pendulum velocity. This procedure may be open to criticism on the basis that deformation of the pendulum may prevent the rate of strain from being equal to the pendulum velocity. It is believed that the motion of the center of mass of the pendulum is not appreciably affected by this second order deformation, and the accuracy of the results substantiates this statement. Work is now in progress to determine experimentally the actual deformation of the specimen during impact.

When the dynamic force-elongation diagram is plotted, it is found that the slope up to the yield point is less than that determined statically. This discrepancy may be due to elasticity in the mechanical parts of the test set-up as mentioned in the previous paragraph. With the exception of the diagram for S.A.E. No. 6140 steel, the elastic line in the beginning of the force elongation curves was drawn with a slope according to Hooke's law. This same procedure has been followed by Honegger[2] in his work on tension impact of hard and annealed copper and aluminum.

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[2] E. Honegger, "Schlag-Zerreissversuche an Al und Cu," *Bericht No. 95*, Eidgenossische Materialprüfungsanstalt an der E.H.T. in Zurich (1937).

EXPERIMENTAL RESULTS

The materials given in Table II were used in this investigation A representative force-time diagram for each of the materials is shown in Fig. 4. The force-elongation diagrams, resulting from an analysis of the corresponding force-time diagrams, are shown in Figs. 5 to 12.

In addition to the dynamic tests of these materials, the static force-elongation diagrams were determined in the usual manner. The minimum diameter was measured for each increment of load. The results of these tests are shown in Table II and in Figs. 5 to 12. For purposes of comparison with the results obtained by H. C. Mann,[3] "potential force-elongation curves" were determined. In computing these curves, the forces for each increment of deformation are multiplied by the ratio of initial area to the least area of the specimen for each load increment.

The average values from these tests are reported in Table II. The comparative ratios of the average values of the physical properties under dynamic and static conditions are given in Table III.

It will be seen from these tables and the corresponding curves that the yield forces, indicated by the abrupt change in slope of the curves under dynamic conditions, are from 20 to 52 per cent higher than those under static conditions. The maximum dynamic forces are from 9 to 40 per cent higher than the maximum static forces. S.A.E. No. 6140 steel is an exception to this last statement, in that the maximum forces are the same for both types of loading. This material shows no definite yield point.

In the tests on S.A.E. Nos. 6140 and 1015 steels, brass, aluminum, and copper, the dynamic energies are 20 to 186 per cent higher than the energies given by the "potential force-elongation curves." These results are not in agreement with those reported by Mann.[3] This may indicate that the relation between the tension static and dynamic tests is more complicated than one might expect from the results presented so far by Mann.

The dynamic and potential energies for the S.A.E. No. 1018 steel agree with each other to within 4 per cent. With 18 per cent chromium, 8 per cent nickel stainless steel and 17S-T duralumin, the dynamic energies are, respectively, 40 and 12 per cent less than the potential energies. This may be due to the fact that the critical velocity of these materials is below the impact velocity of 11.3 ft. per sec. used in this investigation.

TABLE III.—RATIOS OF DYNAMIC AVERAGE TO STATIC AVERAGE VALUES.

Material	Yield Point	Maximum Load	Elongation	Reduction of Area	Energy
S.A.E. No. 6140[a]	......	1.020	2.205	2.940	2.865
S.A.E. No. 1015[b]	1.328	1.285	1.418	1.055	1.376
S.A.E. No. 1018	1.995	1.397	0.992	0.946	0.964
18-8 Alloy	1.224	1.212	0.682	0.782	0.600
Duralumin 17S-T	1.294	1.094	1.000	1.035	0.885
Brass[b]	1.387	1.142	1.163	1.136	1.203
Aluminum	1.522	1.323	1.628	1.093	1.627
Copper	1.433	1.390	1.527	0.986	1.597

[a] Oil quenched from 1620 F.
[b] Cold-rolled.

For S.A.E. Nos. 6140 and 1015 steels, brass, aluminum, and copper, the elongations measured on the dynamic specimens are from 16 to 120 per cent greater than those measured on the static specimens. In the case of S.A.E. No. 1018 steel and 17S-T duralumin, the elongations are the same under both types of loading. For 18 per cent chromium, 8 per cent nickel stainless steel, the dynamic elongation is 32 per cent lower than the static elongation.

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[3] H. C. Mann, "The Relation Between the Tension Static and Dynamic Tests," *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part II, p. 323 (1935).

The reduction of area for S.A.E. Nos. 6140 and 1015 steels, brass, and aluminum is from 9 to 191 per cent larger dynamically than statically. The difference between the static and dynamic values of reduction of area is less than or equal to ±5 per cent for specimens of S.A.E. Nos. 1015 and 1018 steels, 17S-T duralumin, and copper.

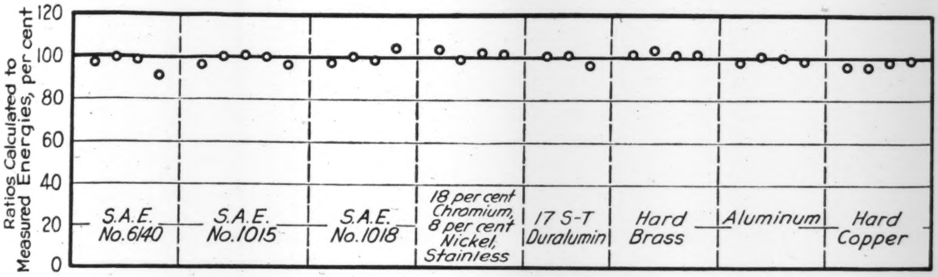

FIG. 13.—Comparison Between Calculated and Measured Energies.

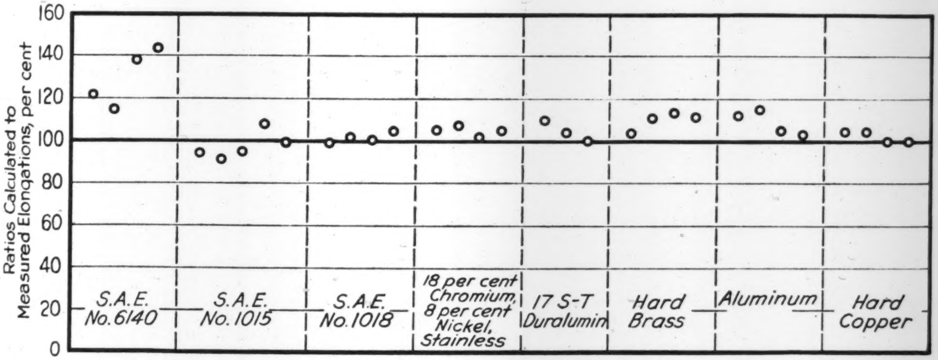

FIG. 14.—Comparison Between Calculated and Measured Elongations.

These results seem to indicate that those materials which have been cold worked will have better dynamic properties than would be expected from the static tests.

An examination of Table II will show that the energies obtained from the dynamic force-elongation diagrams check very closely with those given by the readings on the impact machine. The values of the energies obtained from the curves, expressed in percentage of the pendulum values, are shown in Fig. 13. In Fig. 14 is given the percentage of the computed to the measured values of elongation. It can be seen that the agreement of these two quantities establishes very satisfactorily the validity of the method developed in this research.

SUMMARY

In brief, the results of this investigation can be summarized as follows:

1. A method has been developed for the determination of force-elongation diagrams for metals under tension impact loads; and the reliability of this method has been demonstrated by the agreement shown between the measured and computed values of energy and elongation.

2. The dynamic force-elongation dia-

grams for several materials have been determined and compared with the static diagrams of the same materials. In most of the cases considered, it has been found that the yield and maximum forces are higher with dynamic loading than with static loading.

3. There is some indication that the physical properties of cold-worked materials in dynamic tension are superior to those in static tension. These cold-worked materials show considerably more elongation, and in some cases greater reduction in area, when loaded dynamically than when loaded statically.

4. The results obtained with 18 per cent chromium, 8 per cent nickel stainless steel and 17S-T duralumin indicate that these two materials may possess very low critical velocities.

Acknowledgments:

The authors wish to express their appreciation to the sponsors of this program, who consist of Allis-Chalmers Manufacturing Co., Caterpillar Tractor Co., General Petroleum Corporation of California, Hughes Tool Co., Lane-Wells Co., National Supply Co., Arnold Pfau, A. O. Smith Corp., Union Oil Company of California, and W. M. White, for permission to publish the results of these investigations. Appreciation is also extended to Theodore von Karman, of the California Institute of Technology, for his valuable assistance and criticism in this work; and to F. J. Converse, of the same institute, for his suggestions; and to E. E. Simmons for his very helpful work in the construction of the electrical equipment.

DISCUSSION

Mr. T. McL. Jasper[1] (*presented in written form*).—This paper comes as another contribution to the subject of tensile shock loading of material and promises to help us in the very difficult matter of selecting materials to sustain shock in service.

Several designing and testing engineers have for some time felt that the notched bar impact bending test did not contribute very materially toward giving reliable information for the selection of materials to use in locations where shock is to be a matter of importance. I would not like to say that the so-called notched-bar impact bending test is useless, but it is felt that some of the difficulties associated with the results obtained are due to factors which allow results to vary so illogically with slight changes in dimensions and notch shape. A study of the amount of metal affected in the notched-bar impact bending specimen by deformation may give a clue to some of the erratic results. It has been frequently noticed by the writer that the wedge shaped dimensions of the affected zone near the fracture will vary considerably in angle as the width of such specimens are varied in the same materials and treatment. The variation of the notch shape also has a similar effect. This leads one to arrive at the conclusion that such results when used must be used with care and discrimination because it is felt that such a test does not even allow us to arrive directly at energy absorption quality nor the notch sensitivity of materials in dynamic service.

For the above reason the writer is happy to encourage the hope that the impact tension test with variations such as Clark and Dätwyler, and Mann have introduced may become a profitable direction in which to go to discover a serviceable test for selecting materials for dynamic loading and to discover the effect of notching as well.

I should also like to draw attention to the work of recent years which has used the fatigue machine to arrive at notch effect in steels. The test is quite cumbersome of application, however, but there is no doubt but that the logic of the results bears directly on rotating equipment which has to accommodate service notches. I would also like to point out that the notched-bar impact bending test does not correlate with these notched fatigue specimen results.

Of course what is desired is a simple test similar to the torsion or tension test which will have the engineer and the metal producer satisfied that the results bear directly on the desired service quality of shock absorption or notch sensitivity.

Messrs. H. C. Mann[2] and R. K. Haskell[2] (*presented in written form*).—This paper presents a novel method for obtaining force-time measurements in the tension impact test. There are several debatable conditions involved, however, which require further consideration and verification before the results can be conclusively accepted.

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[1] Director of Research, A. O. Smith Corp., Milwaukee, Wis.

[2] Senior Materials Engineer, and Captain, Ordnance Dept., U. S. Army, respectively, Watertown Arsenal, Watertown, Mass.

The Watertown Arsenal laboratory has naturally been intensely interested in the behavior of materials under dynamic loading and in cooperation with the electrical and mechanical sections of the Massachusetts Institute of Technology, has thoroughly studied the possibilities for determining the actual stress conditions involved in the tension impact test. From these studies, it was evident that in any method employed, the natural frequency of the devise used should be several times the period increment of the least reading desired.

In the work presented by the authors, it is stated that "The linear relation between the impact forces and the dynamometer deformations is valid as long as the period of the natural vibration of the dynamometer bar is short compared with the duration of impact." In the tension impact test, the governing factor is not the duration of impact but rather the rate of change of the applied load. Under these conditions, assuming a modulus relationship up to 40,000 lb. per sq. in. the corresponding elongation over a 1-in. gage would be approximately 0.0013 in. At a velocity of 11.3 ft. per sec., the time to reach the yield zone would be of the order of 0.00001 sec. The natural frequency of the dynamometer is stated as 16,000 cycles per second or 0.00003 sec. per one-half period, from which it is clearly evident that reactions are taking place at a rate several times faster than the natural frequency of the dynamometer. It is obvious under these conditions that the dynamometer is not recording the force of rupture but may be in error as much as several hundred per cent.

A further point which is not considered entirely satisfactory is the fact that the dynamometer was calibrated under static and not dynamic conditions. Under dynamic loading, where force applications are at a rate greater than the natural frequency of the bar, waves of varying intensity will be caused to travel along the bar in such manner that the values obtained may not represent true conditions.

It is significant to note from similar experiments at the National Physical Laboratory, England, using piezo-electric crystals, that from early experiments (1932) using a gage of relatively low natural frequency, curves similar to those noted in this paper were obtained. However, in 1935, a piezo-electric gage of very high natural frequency was obtained from which the load-elongation diagram in the yield zone was found to be essentially the same under dynamic as under static conditions.

In this paper, no details as to specimen form and dimensions or the number of tests are given, although considerable variation between the static and dynamic ductility and energy values is reported. In view of the fact that the Watertown Arsenal has tested most of the materials listed under both static and variable velocity conditions, and in no case have these values differed more than would normally be expected in a series of static tests of the same material, the validity of the results presented is seriously questioned. As pointed out by Haskell,[3] to obtain a correlation between the static and dynamic tests, it is absolutely essential that the work of rupture be confined entirely to the reduced section. This necessitates square-end shoulders of sufficient section that no deformation in them can occur. The gage section must also be tapered slightly toward the middle to eliminate any possibility for double necking. Certain definite precautions must be taken in obtaining the data for the potential curve, otherwise the correlation between the potential curve and the dynamic test does not hold. The wide differences between the values as reported by the authors leads one to conclude that the proper precautions were not observed. The investigations at Watertown Arsenal have never revealed a difference between the energy from the potential curve and the dynamic energy from the Charpy tension impact test provided the transition velocity is not exceeded. In the list of materials tested, only the 18 per cent chromium, 8 per cent nickel alloy has a transition velocity below the impact velocity used by the authors.

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[3] R. K. Haskell, "True Stress-Strain Curves, for Polycrystalline Material," *Journal of Applied Physics*, Vol. 9, No. 1, January, 1938, p. 30.

As to the materials selected for this work, it has been our experience that a steel in the as-quenched condition will invariably produce erratic and unreliable results, a fact which may explain the extreme differences reported. Also certain of the aluminum compositions have been erratic unless in the fully stabilized condition.

In the carrying on of any research program on impact, it is absolutely essential to have the material tested in such a metallurgical state that the same test repeated will give, within the limits of probability, identical results. At Watertown Arsenal it has been found that, below the transition velocity, the ductility of the static and dynamic tests are identical. The values reported by the authors do not in general agree. Some factor not evident is upsetting this relationship. It could be either metallurgical or difference in geometry of the static and dynamic test specimen or some other factor not apparent to the investigators.

Until such time as these debatable points have been fully verified and proven satisfactory, the data presented can only be considered in the light of an interesting experiment warranting further study and corroboration.

MR. L. B. TUCKERMAN.[4]—We shall know more about the meaning of the impact test when we learn more about the details of the sequence of events during an impact test. Today we have hopes of really understanding these details, which we did not have before the day of high-speed cameras, vacuum tube amplifying circuits, and oscillographic methods of recording phenomena which take place in incredibly short periods of time. Clark and Dätwyler's ingenious method of adapting some of these modern tools to the study of the impact test have increased these hopes. The results they have already obtained are significant and give rise to the hope that by modifications of their methods or the use of others we shall learn still more.

I hope that they will continue their study and in particular hope that the free frequency of their dynamometer can be materially increased.

The oscillographic curves reproduced show high frequency vibrations superimposed upon the general trend. With apparatus responding to higher frequencies I feel sure these would have been considerably larger. In the analysis they are ignored. This is not in the way of criticism. With the limitations of the apparatus that was necessary.

Are these fluctuations a significant factor in these tests? At present we have no means of knowing, but some phenomena observed in brittle materials subject to impact indicate that the character of the high frequency vibrations set up by the shock materially influences their resistance to breakage.

It will not be easy to devise means to answer such questions. The free frequency of their specimens in longitudinal vibration will be approximately 100,000 cycles per second.

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[4] Assistant Chief, Division of Mechanics and Sound, National Bureau of Standards, Washington, D. C.

To determine whether the shock waves travelling up and down the specimen materially affect the results would require a dynamometer which would respond accurately to frequencies of this order. This would certainly be difficult but perhaps is not altogether hopeless.

As you can see all my criticism of this paper is in the nature of "wishful thinking." The authors have done so well that I am hoping they will continue and do much more.

MR. M. F. SAYRE.[5]—The paper by Messrs. Clark and Dätwyler carries on to a further degree work which has already been done by others, as, for instance, rather notably by Winlock and Leiter in their studies in which they have measured the variations in stress-strain curves at moderate speeds.[6] Their figures also show with increasing speed an increase in total energy absorbed, pointing in the same direction as the work done by Clark and Dätwyler. As stated by others, the work reported in the present paper is very important as a start. I strongly urge that the authors go on further, check the values, change the method slightly, attempt to verify it and then later report further on their results.

MR. G. DÄTWYLER.[7]—It has indeed been gratifying to receive all this constructive criticism and the various suggestions presented.

The authors in preparing the paper intended to report on what may be called the first step so far achieved in this research, fully realizing that some allowance has to be made as to the response of the dynamometer in the elastic range of the diagrams. But Mann's statement that "the dynamometer . . . may be in error by as much as several hundred per cent" is evidently somewhat too strong. An elementary law of dynamics of ideal elastic bodies states that in the extreme case of an infinitely fast applied load the elastic deformation will be twice the static value under the same load. With damping which is always present this "overshooting" is reduced to less than the theoretical maximum of 100 per cent.

It is felt that the elastic range of deformation would have to be the subject of a special research, in order to study phenomena such as mentioned by Tuckerman. But this phase of the impact problem has so far not been the primary interest in the present research program. And if, as Mann reported, the work conducted in England has shown that the diagrams in the yield zone are essentially the same under both static and dynamic conditions, then the plastic zone becomes all the more important.

As stated in the paper, the specimens had a gage length of 1 in. and a diameter of 0.20 in., within the limits of ± 1/2000 in. The gage section was cylindrical. The authors have found that at the low speeds used in this research, tapered specimens were not necessary. In high-speed work (30 to 260 ft. per sec.) that was carried out about three years ago it was found that double necking occurred.

The transition from the gage section to the shoulders was square, with a radius of fillet of about $\frac{1}{64}$ in. The ends were threaded with S.A.E.—20 threads per inch with a root diameter of 0.43 in. Naturally the specimens for the two types of test were, for each material, taken from the same bar. The results obtained within the various groups of specimens were very consistent. Five dynamic and three static specimens were used for each material. During the development work several hundred specimens of hot-rolled and cold-rolled mild steel in the as-rolled condition were broken. Consistently the hot-rolled material gave results that were in complete agreement with Mann's relation, whereas the cold-rolled material consistently gave about 42 per cent more elongation, and 38 per cent higher energy values, dynamically than statically. The agreement with Mann's relation obtained with the hot-rolled mild steel makes it hard to understand why "some factor not evident" should upset this relationship and thus make the validity of the results presented seriously questionable.

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[5] Professor of Applied Mechanics, Union College, Schenectady, N. Y.

[6] Joseph Winlock and Ralph W. E. Leiter, "Some Factors Affecting the Plastic Deformation of Sheet and Strip Steel and Their Relation to the Deep Drawing Properties," *Transactions*, Am. Soc. Metals, Vol. 25, No. 1, March, 1937, pp. 163-184.

[7] Research Fellow in Aeronautics, California, Institute of Technology, Pasadena, Calif.

THE THEORY OF IMPACT TESTING: INFLUENCE OF TEMPERATURE, VELOCITY OF DEFORMATION, AND FORM AND SIZE OF SPECIMEN ON WORK OF DEFORMATION[1]

By D. J. McAdam, Jr.,[2] and R. W. Clyne[3]

Synopsis

An impact test should not be viewed as a simulation of shock in service, and should not be limited to metals that are to be subjected to impact in service. The object of an impact test should be to determine whether a structure or machine would be in danger of failure from brittleness under service conditions.

In most structures and machines, there are one or more regions of relatively high concentration of three-dimensional tensile stress. In these regions, during application or change of load, the specific velocity of deformation is relatively high. If, as a result of this increased velocity of deformation, the resistance to flow becomes relatively higher than the resistance to cohesive failure, the fracture will be brittle rather than ductile in character, and the amount of energy required to produce failure will greatly decrease. Similarly, a decrease in temperature results in an increase in the resistance to flow in relation to the resistance to cohesive failure and so causes danger of this same granular fracture (the so-called cold brittleness) rather than fibrous fracture. The energy required to produce fracture decreases very rapidly (in most steels) on passing a certain temperature, which is dependent upon the composition and heat treatment of the metal, the shape and size of the piece, and the speed of loading.

Anything which tends to raise this critical temperature increases the danger of cold brittle failure, and *vice versa*. Thus, the greater the size of the structure, the greater is the tendency to cold brittleness, and the less is the specific work of cold-brittle steel. This paper discusses the problem of impact failure as affected by these different variables.

(1) Introduction

Although the behavior of metals under impact, and the significance of impact tests have been the subject of much investigation, confusion is rather prevalent as to the fundamental factors affecting the energy that metals will absorb under rapidly applied stresses, and as to the influence of form and size of specimen on the total work. The object of this paper is to illustrate and discuss the influence of these fundamental factors on total work.

During the past 15 yr., information

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[1] Published as a personal contribution by the authors, by permission of the Director of the National Bureau of Standards. An abridgment of a chapter of a book by the authors entitled "Strength of Metals: With Special Reference to Spring Materials and to Stress Concentration." The book is to be published shortly under the auspices of the Special Research Committee on Mechanical Springs of the American Society of Mechanical Engineers.

[2] Metallurgist, National Bureau of Standards, Washington, D. C.

[3] Sales Engineer, American Steel Foundries, Chicago, Ill.

obtained by various investigators has thrown much light on this subject, and has shown that ductility and total work are dependent upon the relationship between resistance to plastic deformation and resistance to fracture. If in any given case the "flow stress" or tensile stress required to produce plastic deformation is less than the resistance to fracture, failure will be accompanied by plastic flow, and a relatively large amount of energy will be absorbed. If, however, the flow stress, or resistance to plastic deformation is greater than the resistance to fracture, a brittle fracture will result, accompanied by a relatively small absorption of energy.

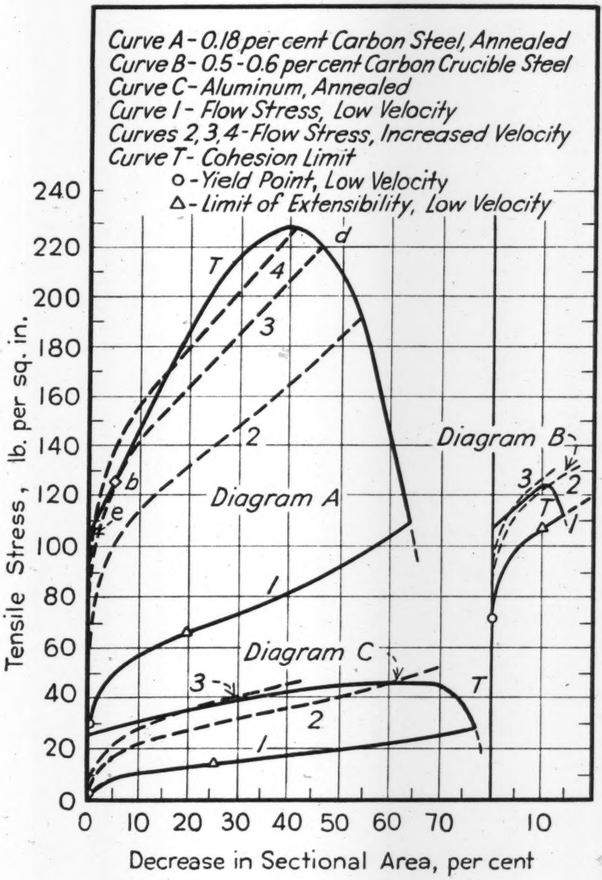

Fig. 1.—Variation in Cohesion Limit and Flow Stress, with Plastic Deformation.

By "resistance to fracture" is meant, not the interatomic forces, but the technically determinable stress necessary to cause fracture. An example of such technically determinable stress is the "true tensile stress" at fracture, the breaking load divided by the sectional area. In an ordinary tension test, however, the fracture usually occurs after more or less plastic extension. The resistance to fracture thus measured, therefore, is not the resistance of the original metal, but of metal that has been plastically deformed during the test. By such deformation, resistance to fracture is greatly altered.

More than ten years ago, Ludwik (**13, 14**)[4] pointed out the need for knowledge of resistance to fracture in absence of plastic deformation. Since then, considerable information on this subject has been obtained, due largely to the work of Kuntze (**11, 12**). The resistance to fracture of ductile metal, without plastic deformation, may appear at first sight to be of merely academic interest. Ductility is generally considered to offer assurance that metal will not fail without plastic deformation. Under some conditions of service, however, high natural ductility gives no assurance against fracture with little or no plastic deformation. These conditions tend to cause resistance to plastic deformation to be high in relation to resistance to fracture. If conditions are such that the resistance to plastic deformation is equal to or greater than the resistance to fracture, the metal fails without plastic deformation. If the initial resistance to plastic deformation is less than the resistance to fracture, the metal can deform until the increasing resistance to deformation becomes equal to the resistance to fracture.

(2) FACTORS AFFECTING THE RELATIONSHIP BETWEEN RESISTANCE TO PLASTIC DEFORMATION AND RESISTANCE TO FRACTURE

The relationship between resistance to plastic deformation and resistance to fracture depends not only on the metal but also on the degree of prior plastic deformation, on temperature, velocity of deformation, combination of stresses, and form and size of specimen. The influence of prior plastic deformation on resistance to plastic deformation, resistance to fracture, and the relationship between the two, is illustrated by the diagrams in Fig. 1, which is based on data published by Kuntze (**12**). Curve *T* of diagram *A* represents the variation of the technically determined "cohesion limit" of an 0.18 per cent carbon steel, with plastic deformation. Curve 1 represents the variation of the flow stress (true tensile stress necessary to cause or continue plastic deformation) with plastic deformation. The curve of resistance to fracture first rises, then descends. The curve of flow stress rises continuously. The point of intersection, where the flow stress equals the cohesion limit, represents the point of fracture.

The greater the velocity of deformation, the higher is the curve of flow stress. Curve 1 represents the variation of flow stress under slowly applied load. Curves 2, 3, and 4, added by the authors, represent similar variation under successively higher velocities. As here illustrated, the greater the velocity of deformation, the higher is the flow stress in relation to the cohesion limit, and the less is the ductility. Diagrams *B* and *C* represent similar relations for a steel of higher carbon content and for aluminum.

Elevation of the curves of flow stress is indicated as due to increase in velocity of deformation; such elevation however could as well result from an increase in the ratio of the three-dimensional tensile stress to the longitudinal stress (the spatial stress ratio). Spatial (three-dimensional) stress is induced by a notch in a specimen under tension or bending (not under pure torsion). The influence of a notch on total work, as shown by Kuntze (**12**) and others, generally depends much more on the induced spatial stress than on the stress concentration. The deeper and sharper the notch, the higher is the spatial stress ratio, and the higher is the ratio of flow stress to shear stress. Consequently, the higher the spatial stress ratio, the higher is the corresponding curve of variation of flow stress with plastic deformation, in relation to the curve of variation of the cohesion limit.

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[4] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 131.

Elevation of the curve of flow stress in relation to the curve of cohesion limit is also caused, in some metals, by decrease of temperature. Temperature, probably unlike velocity of deformation and spatial stress ratio, affects both resistance to plastic deformation and resistance to fracture, but it generally affects these variables differently, and thus generally affects their interrelationship and the work of deformation to fracture.

(3) INFLUENCE OF TEMPERATURE ON THE TOTAL WORK OF CARBON STEELS

Of the variables that affect the relationship between resistance to plastic deformation and resistance to fracture, the one that should receive first detailed consideration is temperature. The influence of any other significant variable may then be studied with reference to the relationship between temperature and total work.

The influence of temperature on total work varies greatly with the metal. Temperature has great influence on the total work of carbon steels and the other heat-treatable steels. Low-carbon steels are especially sensitive to the influence of temperature. This is illustrated in the upper diagram of Fig. 2, which is constructed from data in a paper by Greaves and Jones (8). The curve here shown illustrates the effect of temperature on the notched-bar impact value for a steel containing 0.18 per cent carbon. The prominent features of this curve are the steep drop in impact value at low temperature, the sharply defined minimum at 1075 F., and the steep rise to a maximum at slightly higher temperature.

The most striking of these features is the steep drop at low temperature. Corresponding to this abrupt change in impact value, there is change from fibrous to granular fracture. Over a small range of temperature in the vicinity of the low-temperature drop, the impact values tend to be either concordantly high or concordantly low.

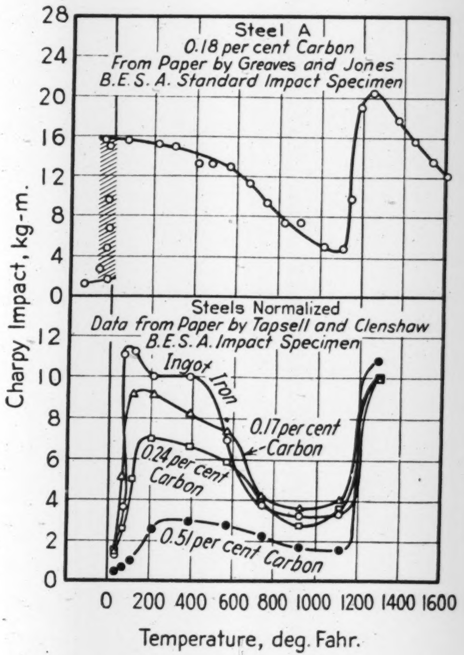

FIG. 2.—Influence of the Temperature of Steels on Total Work.

The influence of carbon content on the form and position of the impact-temperature curve is illustrated in the lower diagram of Fig. 2, which represents data published by Tapsell and Clenshaw (25, 26). The curves here shown represent normalized steels with carbon content ranging from that of ingot iron to 0.51 per cent. With increase in carbon content, the low-temperature drop becomes less steep and extends over a wider temperature range. The temperature of the bottom of the drop evidently is little affected by carbon content. This temperature, however, is considerably higher than in the upper diagram. The positions of the low-temperature drop and the high-temperature minimum are greatly influenced by a number of variables, which are considered later.

The low-temperature drop in impact value, represented by a line in the lower diagram of Fig. 2 and in following figures, might more properly be represented by a band, as in the upper diagram of Fig. 2. The abrupt drop in impact value represents a discontinuous transition from fibrous to granular fracture. Even through the line may descend gradually, as does the line representing 0.51 per cent carbon steel in Fig. 2, discontinuity of the transition is indicated by the appearance of the surface of fracture. Within the transition range of temperature, the fracture consists of alternating fibrous and granular areas. With descent along the sloping line, the proportion of granular areas increases until the entire fracture is granular. The discontinuity of the transition from fibrous to granular fracture is not due to instability of the metal, but to instability in the relationship between resistance to plastic deformation and resistance to fracture.

The cause of the abrupt variations in impact value (illustrated in Fig. 2) is the effect of temperature on the relationship between flow stress and cohesion limit. The decrease in total work of steel at low temperature may be explained on the assumption that the rate of increase in flow stress with fall of temperature is greater than the rate of increase of the cohesion limit.

(4) Influence of Temperature on the Tensile Elongation of Steels

Total work obviously depends on both strength and ductility. Steels arranged in the order of their ductility, however, usually are found to be arranged in the order of total work. The influence of temperature on the ductility of steels, therefore, is qualitatively similar to the influence of temperature on total work. Static tensile elongation values, accordingly, are presented in Figs. 10 and 11 as evidence of the qualitative influence of temperature on the total work of slowly loaded, unnotched specimens.

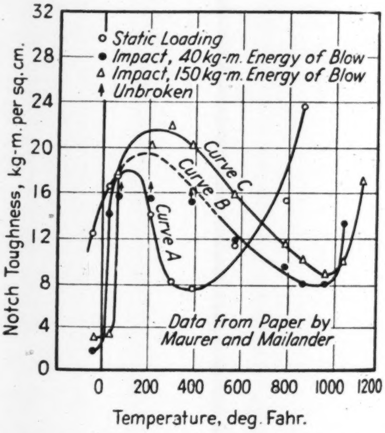

FIG. 3.—Influence of Velocity of Deformation on the Curve of Variation of Total Work with Temperature.

The rapid decrease in ductility with fall of temperature from the low-temperature maximum, illustrated in Figs. 10 and 11, evidently is similar in nature to the low-temperature drop in absorbed energy illustrated in Figs. 2 to 5. In position and slope, however, the low-temperature descent in Figs. 10 and 11 differs greatly from the low-temperature drop in Figs. 2 to 5. This difference in position and slope is due to difference in the spatial stress ratio and in the specific velocity of deformation. The higher the spatial stress ratio and specific velocity of deformation, the higher is the temperature of the transition from fibrous to granular fracture.

(5) Influence of Velocity of Deformation on the Total Work of Carbon Steels

Any variable that affects the relationship between flow stress and cohesion limit affects the form and position of the curve of variation of total work with temperature. One of the most important of such variables is velocity of deformation. The influence of velocity of deformation on the curve of variation of total work with temperature is illustrated by Fig. 3, which is constructed from data obtained by Maurer and Mailander **(17)**. Curve *A* represents results obtained with static loading. Curves *B* and *C* represent results obtained at two different momenta of impact, and hence at two different velocities of deformation. With increase in the velocity of deformation, the low-temperature drop in total work evidently moves to higher temperature. This change in position is due to the fact that increase in velocity, like decrease in temperature, raises the curve of flow-stress with reference to the curve of cohesion (Fig. 1).

The high-temperature minimum of total work under slow loading is at about 400 F. With change from slow loading to impact at relatively low momentum, this minimum moves to much higher temperature. Further increase in momentum of impact (velocity of deformation), as illustrated by curve *C* of Fig. 3, moves the minimum to only slightly higher temperature. The high-temperature minimum of curve *C* is at about the temperature of the corresponding minimum of the curves in Fig. 2.

Under slow loading, the temperature of this minimum is the temperature at which carbon steels have maximum strength and minimum ductility. Because of the blue "temper color," which steel tends to assume at about 450 F., the low ductility at this temperature has long been known as "blue brittleness." As shown in Fig. 3, however, the temperature of minimum absorbed energy varies greatly with the velocity of deformation. A better name for this property, therefore (as pointed out by Maurer and Mailander), is "hot brittleness."

(6) Relation of Hot Brittleness and Cold Brittleness to the Age-Hardening of Steel

The hot brittleness of steels was shown by Fettweis **(6)** to be a manifestation of the long-known "age-hardening" of steel. Similar views were expressed independently by Archer **(1)**. Age-hardening is most prominent in low-carbon steels, especially in steels that have been cold worked. An immediate effect of cold work is the usual increase in yield point and tensile strength, and decrease in ductility. After considerable time at room temperature or after a short time at elevated temperature, however, there is considerable further increase in strength and decrease in ductility. These secondary changes are known as "age-hardening."

At a temperature of about 450 F., as shown by Fettweis **(6)**, age-hardening of steel is so much accelerated that it is completed during plastic deformation. if the deformation is carried on slowly, At this temperature, therefore, steel (not previously aged) has maximum strength and minimum ductility (blue brittleness). Metal plastically deformed at the elevated temperature and afterward cooled to room temperature is as hard as if it had been plastically deformed to the same amount at room temperature and afterward aged. Completely aged steel, like non-ferrous metals and unlike unaged steel, is said to decrease steadily in strength with rise in temperature.

Hot brittleness, therefore, seems to be due to the age-hardening during the test. As the temperature of hot brittleness (as shown by Fettweis) is the temperature at which the rate of age-hardening equals the rate of deformation, this temperature varies with the rate of deformation in testing. In an impact test of a notched specimen, the specific velocity usually is so high that the temperature of hot brittleness is about 1050 to 1200 F. At exceedingly slow rates of deformation age-hardening as shown by Archer (1) may be completed during deformation at room temperature.

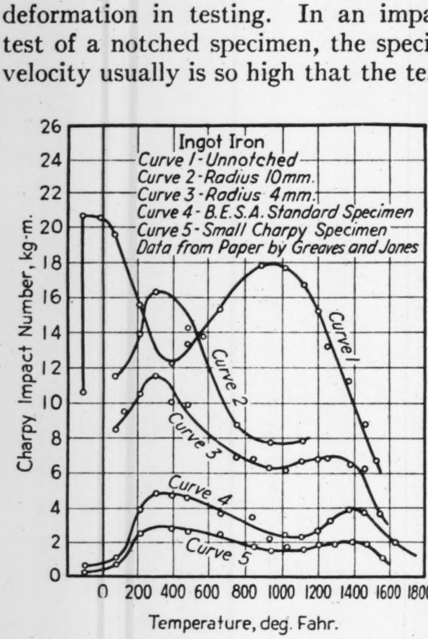

FIG. 4.—Influence of Notches on the Curve of Variation of Total Work with Temperature.

The age-hardening of steel, according to Ludwik (13, 15), is due to precipitation of cementite or other constituents from supersaturated solid solution in the ferrite. The solubility of the dispersed phase probably decreases with fall of temperature. This decrease in solubility, and consequent increase in quantity of the dispersed phase, is believed to be the cause of the rapid rise of the yield point of steel with fall of temperature. As illustration of this unique behavior of steel, Ludwik (15) states that the yield point of low-carbon steel is many times as great at the temperature of liquid air as at ordinary room temperature, whereas aluminum and copper show no such effect of temperature. As the "true breaking stress" of low-carbon steel was found to be little greater at liquid air temperature than at room temperature, the ratio of yield point to cohesion limit evidently increases rapidly with fall of temperature. Ludwik's theory, therefore, evidently relates both cold brittleness and hot brittleness of steel to precipitation hardening.

Plastic deformation, because of its tendency to accelerate transformation from a metastable to a stable condition, accelerates the deposition of the dispersed phase. Rise of temperature, although it has a similar effect on the dispersed phase, also increases the solubility. The fact that plastic deformation accelerates deposition of the dispersed phase without increasing the solubility, may account for the importance of plastic deformation in age-hardening.

(7) INFLUENCE OF NOTCHES ON THE RELATION BETWEEN TOTAL WORK AND TEMPERATURE (CARBON STEEL)

The influence of notches on the relation between total work and temperature is illustrated by Fig. 4, which is based on data obtained by Greaves and Jones (8) with ingot iron. Curve 1 represents results obtained with unnotched specimens; the other curves represent results obtained with specimens having notches of various forms. The greater the depth and sharpness of the notch, the lower is the corresponding curve in the diagram. The lowering of the curve of total work, when caused by a notch, is due to decrease in the volume of plastically deformed metal.

In lateral location, moreover, curve 1 differs considerably from the curves representing notched specimens. Especially noteworthy is the difference in the temperature of hot brittleness. For the unnotched specimens, this temperature is about 400 F.; for the notched specimens, the temperature is about 950 to 1050 F. Although all these specimens were tested by impact, hot brittleness of the unnotched specimens was at about the usual temperature of hot brittleness under slow loading. The volume of deformed metal in the unnotched specimens, therefore, probably was so great that the specific velocity of deformation was relatively low. With notched specimens, the specific velocity evidently was so great that hot brittleness was encountered at a much higher temperature, the temperature at which hot brittleness is usually encountered in impact testing.

The low-temperature drop in total work of unnotched specimens represented in Fig. 4 is at about −110 F. Even the bluntest notch moved this drop to much higher temperature. The sharper the notch, the higher is the temperature of the drop; the sharpest notches represented in the figure gave low impact values at ordinary room temperature. This effect of a notch on the temperature of transition from fibrous to granular fracture is similar to the effect of increase in the velocity of the testing machine (Fig. 3). A notch, like an increase in the velocity of the testing machine, raises the flow stress with reference to the cohesion limit. This effect of a notch is due in part to the induced spatial stress, in part to the decrease in the volume of deformed metal and consequent increase in the specific velocity of deformation.

(8) INFLUENCE OF THE BREADTH OF A NOTCHED SPECIMEN ON THE TOTAL WORK OF CARBON STEELS

An encircling notch in a cyclindrical specimen under tension has been shown to cause radial tensile stress. For an impact-bend test, however, the specimen usually is of square or rectangular cross-section and is notched on only one side. While such a notch in a specimen under longitudinal tension causes lateral tensile stress in one direction, the unnotched sides offer opportunity for contraction (in accordance with Poisson's ratio) in the other lateral direction. Because of this contraction along the notch, the spatial-stress ratio tends to be less in such a specimen than in a specimen with encircling notch. The greater the breadth of a specimen notched on only one side, however, the greater is the hindrance to contraction along the notch, and the greater is the spatial-stress ratio. Increase in the breadth of the specimen, therefore, is similar in effect to increase in the depth or sharpness of the notch.

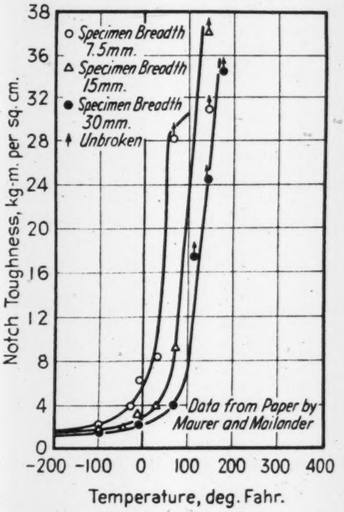

FIG. 5.—Influence of the Breadth of a Notched Specimen on the Temperature of Cold Brittleness.

The effect of breadth of specimen on the position of the low-temperature drop in total work is illustrated by the curves in Fig. 5, which represent data obtained by Maurer and Mailander **(17)**. The curves there shown represent results of impact tests of notched specimens with breadths of 7.5, 15 and 30 mm. Increase in specimen breadth, like increase in the depth or sharpness of a notch, evidently raises the temperature of the steep drop in total work. At any one temperature within this range, the variation in impact value with breadth of specimen is very great.

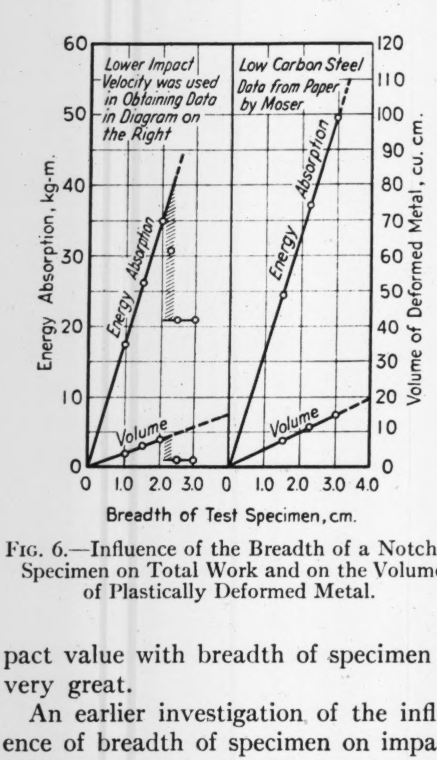

FIG. 6.—Influence of the Breadth of a Notched Specimen on Total Work and on the Volume of Plastically Deformed Metal.

An earlier investigation of the influence of breadth of specimen on impact value was made by Moser **(18, 19)**. As all Moser's tests were made at room temperature, however, his interpretation of the results was hampered by lack of data on the important influence of temperature. Typical results of this investigation **(19)** are shown in Fig. 6. The upper graph in each diagram represents impact values; the lower graph represents corresponding volume of deformed metal.

The total work, as would be expected, evidently varies in accordance with the volume of deformed metal. With impact conditions as represented in the diagram at the left of Fig. 6, impact value and volume increased in proportion to the specimen breadth until the breadth reached 2.0 cm. With further increase in breadth, both impact value and volume dropped abruptly. With specimens 2.5 and 3.0 cm. broad, both impact value and volume were even less than with a specimen only 1.5 cm. broad. This drop in impact value was accompanied by a change from fibrous to granular fracture. With specimens more than about 2.0 cm. broad, therefore, the transition from fibrous to granular fracture would have been at a higher temperature than that of the impact test. Elevation of the temperature of transition with increase in breadth of the specimen has been discussed in connection with Fig. 5.

The diagram at the right of Fig. 6 illustrates the relationship at lowered velocity of impact, the steel and the form of notch being unchanged. At the decreased velocity, the proportionality between breadth of specimen and impact value was maintained to a breadth of at least 3.0 cm. Decrease in velocity, therefore, tends to oppose the embrittling effect of increase in specimen breadth. Decrease in velocity and increase in breadth, as shown in Figs. 3 and 5, have opposite effects on the temperature of the transition from fibrous to granular fracture.

(9) INFLUENCE OF COLD WORK ON THE RELATION BETWEEN TOTAL WORK AND TEMPERATURE (CARBON STEEL)

The influence of cold work on the relation between total work and temperature is illustrated in Fig. 7, which represents data obtained by Greaves and Jones (8) with notched specimens of low-carbon steel. The upper curve in this figure represents hot-rolled material; the other curves represent the same material cold worked to decreases in sectional area of 16 and 33 per cent. Cold work evidently has practically no effect on the position of the high-temperature minimum, but has much effect on the position of the low-temperature drop. With increase in the degree of cold work, the steep drop in total work has been moved from below freezing temperature to ordinary room temperature. Prior plastic deformation, like increase in specific velocity of deformation, evidently raises the temperature of the transition from fibrous to granular fracture.

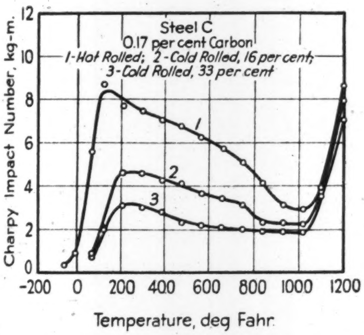

Fig. 7.—Influence of Cold Work on the Curve of Variation of Total Work with Temperature.

The effect of cold work followed by annealing (age-hardening) on the relation between total work and temperature is illustrated in Fig. 8, which represents data obtained by Maurer and Mailander (17). All four curves in this figure represent the energy absorbed in bend tests of notched specimens of low-carbon steel. One pair of curves represents results obtained by slow bending; the other pair represents results obtained by impact. The great influence of velocity on the temperature of hot brittleness, illustrated in Fig. 3, is again illustrated in Fig. 8. This figure also offers additional evidence that the temperature of hot brittleness is little affected by cold work.

The chief purpose of Fig. 8, however, is to show the effect of cold work followed by age-hardening on the temperature of the transition from fibrous to granular fracture. As revealed by the curves representing slow loading, age-hardening moved the transition temperature from about -40 F. to about 30 F. As revealed by the curves representing impact, age-hardening moved the transition temperature from 0 F. to ordinary room temperature. Whether the test be by slow loading or by impact, therefore, age-hardening evidently raises the temperature of the transition from fibrous to granular fracture.

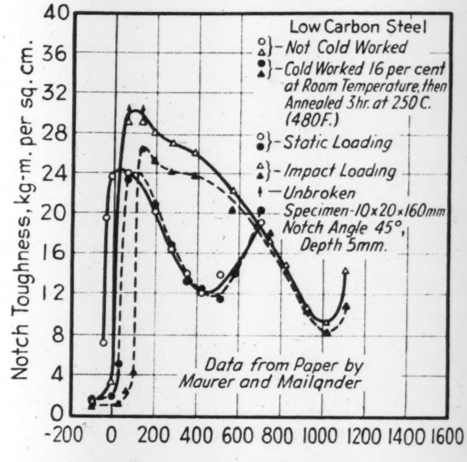

Fig. 8.—Effect of Age-Hardening and of Velocity of Deformation on the Curve of Variation of Total Work with Temperature.

(10) Influence of Heat Treatment on the Relation Between Total Work and Temperature

The influence of heat treatment on the relation between total work and temperature is illustrated in Fig. 9, which represents data obtained by Greaves and Jones (8). The upper diagram of this figure represents results obtained with a 0.45 per cent carbon steel, the lower diagram represents results obtained with a nickel steel. Increase in the strength of steel by heat treatment evidently lowered the temperature of the transition from fibrous to granular fracture. In this respect, therefore, the effect of strengthening by heat treatment is opposite to the effect of cold work. The temperature of hot brittleness evidently is little affected by heat treatment.

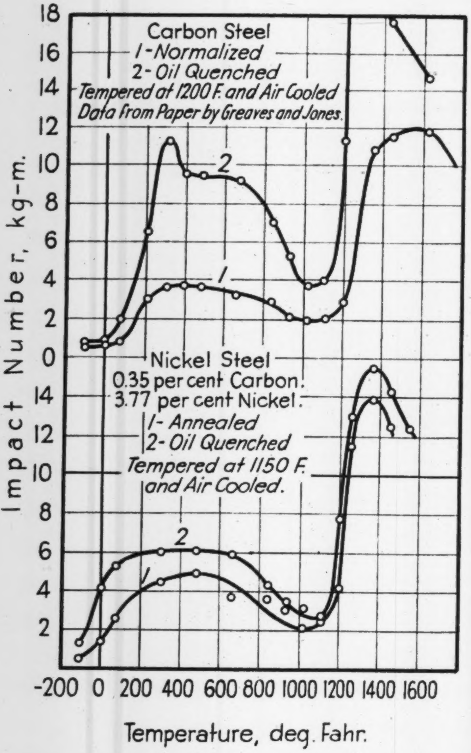

FIG. 9.—Effect of Heat Treatment on the Curve of Variation of Total Work with Temperature.

"Temper brittleness" of some steels, especially chromium-nickel steels, is caused by slow cooling from the tempering temperature. Although steels thus cooled have practically the same tensile properties as steels that have been cooled rapidly from the tempering temperature, impact values at room temperature are much lower for the slowly cooled than for the rapidly cooled steels. A very incomplete picture of this effect of slow cooling, however, is obtained from impact tests made at only one temperature. Comparison of work-temperature curves for slowly cooled and quickly cooled material shows that slow cooling from the tempering temperature raises the temperature of the transition from fibrous to granular fracture. "Temper brittleness," therefore, is merely cold brittleness.

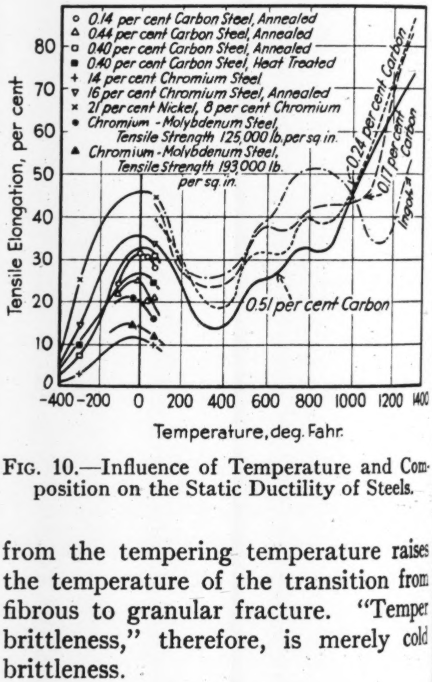

FIG. 10.—Influence of Temperature and Composition on the Static Ductility of Steels.

(11) INFLUENCE OF CHEMICAL COMPOSITION OF STEEL ON THE TEMPERATURE OF COLD BRITTLENESS

The effect of relatively large additions of alloying elements on static ductility at the temperatures of liquid air and liquid hydrogen is illustrated in Figs. 10 and 11. Steels containing 14 and 16 per cent chromium, as illustrated in

Fig. 10, show no evidence of superiority over 0.4 per cent carbon steel at the temperature of liquid air. The order of ductility of these three heat-treatable steels is about the same at low temperature as at ordinary room temperature.

Two impact-temperature curves for a stainless steel containing 0.43 per cent carbon and 12.37 per cent chromium are shown in a paper by Greaves and Jones **(8)**. Each of these curves has a steep drop similar to the low-temperature drop of a curve representing carbon steel, and at about the same temperature. Tendency to cold brittleness, therefore, appears to be no less in a chromium steel of this composition than in a carbon steel. The curves for the chromium steel, however, have no high-temperature minimum, but are nearly horizontal between about 200 and 1300 F. Steel of this composition evidently is not subject to hot brittleness.

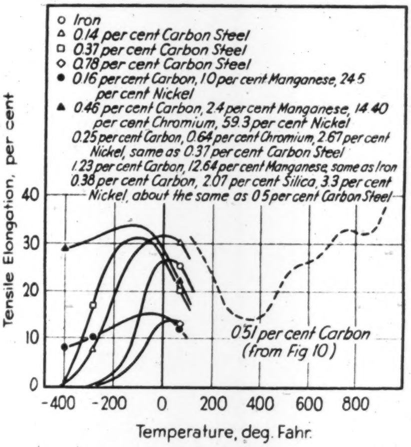

Fig. 11.—Influence of Temperature and Composition on the Static Ductility of Steels.

More recent papers **(5, 24)** have discussed the influence of small percentages of alloying elements on impact resistance at low atmospheric temperatures, such as those encountered in Siberian winters or in aircraft. Carbon steels as rolled or normalized may prove unsatisfactory at these temperatures because of cold brittleness. Suitable heat treatment, by lowering the temperature of the transition from fibrous to granular fracture, decreases the danger of cold brittleness in service. An even greater effect, enhanced by suitable heat treatment, may result from the addition of small percentages of alloying elements. Among the elements that appear valuable for this purpose are chromium, vanadium, silicon, and zirconium. The effect of these elements on the low-temperature drop is illustrated in Fig. 12, representing data obtained by Egan, Crafts and Kinzel **(5)**.

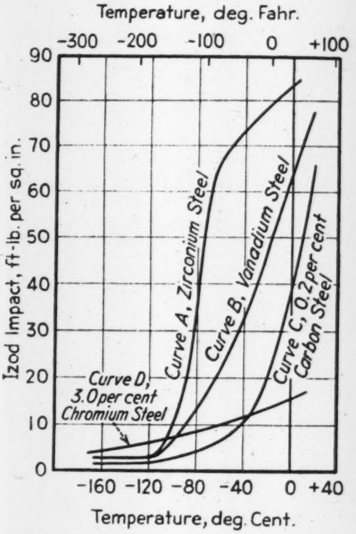

Fig. 12.—Influence of Small Percentages of Alloying Elements on the Temperature of Cold Brittleness.

(12) Influence of Temperature on Ductility and Toughness of Non-Ferrous Metals

Ductility of typical non-ferrous metals at the temperature of liquid air was investigated by DeHaas and Hadfield **(4)**. Copper, nickel, monel metal, and phosphor bronze showed greater ductility at this temperature than at room temperature. Unlike many steels, therefore, these typical non-ferrous metals show no evidence of cold brittleness under slow loading.

The absence of cold brittleness of non-ferrous metals under slow loading does not necessarily mean that cold brittleness is not possible with greater velocity of deformation. With a few exceptions, however, non-ferrous metals show no evidence of cold brittleness even under impact. Impact tests by Greaves and Jones **(9)** revealed no evidence of cold brittleness of any non-ferrous metals except tin and zinc. With both these metals, they found an abrupt change in direction of the impact-temperature curve, probably due to an allotropic change in the metal. Non-ferrous metals not undergoing allotropic change evidently are free from cold brittleness.

Non-ferrous metals, moreover, show no evidence or true hot brittleness. Although these metals show minimum ductility at temperature of recrystallization, this minimum is not similar to the hot brittleness of steels. Hot brittleness of steels has been indicated (Section 6) as due to precipitation hardening during deformation.

(13) REASONS FOR COLD BRITTLENESS OF STEELS AND ABSENCE OF COLD BRITTLENESS OF NON-FERROUS METALS

Reasons for the abruptness of the transition from fibrous to granular fracture of steels have been the subject of much discussion. Although the instability of this transition has led Fettweis **(7)** to conclude that the transition must be attributed to some structural condition of the metal, the authors are of the opinion that both the instability of the transition for steels, and the absence of instability for non-ferrous metals, are explainable with reference to the relative positions of the curves of flow stress and technical cohesion, as illustrated in Fig. 1. An attempted explanation by Kuntze **(11)**, based on this relationship, involves the incorrect assumption that the curve of flow stress, if moved upward, remains parallel to its original position. Actually, however, as illustrated by curves 2, 3, and 4 (added by the authors), elevation of the curve causes the ordinates to increase proportionally, not equally. Because of Kuntze's incorrect assumption, as shown by the authors in a book now in preparation, he fails to give a valid explanation of cold brittleness of steels and absence of cold brittleness of non-ferrous metals.

With elevation of the curve of flow stress in diagram *A* of Fig. 1, representing a low-carbon steel, the point of intersection of this curve with the curve of cohesion moves upward along curve *T*. The abscissa of this point, which represents ductility, gradually decreases. Total work (represented by the area beneath the curve of flow stress), however, gradually increases, up to a certain point. At position 3, the curve of flow stress comes in contact with the ascending portion of the curve of cohesion, at point *b*. Ductility, and total work, therefore, decrease discontinuously from the values corresponding to point *d* to those corresponding to point *b*. This abrupt decrease, consequently, would account for abrupt change from fibrous to granular fracture.

The abruptness of the transition, moreover, is increased because of an accentuating variable, the corresponding abrupt change in the specific velocity of deformation. With discontinuous transition of the intersection from *d* to *b*, there is abrupt decrease in the volume of plastically deformed metal, especially in a notched specimen, and hence abrupt increase in the specific velocity of deformation. This increase in specific velocity causes abrupt further elevation of the curve of flow stress, and thus tends to move the intersection rapidly from *b* toward *e*. The entire curve of flow stress possibly may thus be elevated above the curve of cohesion. The influence of this abrupt increase in specific velocity, therefore, accentuates the abruptness of the transition from fibrous to granular fracture.

For a steel of higher carbon content, as illustrated by diagram *B* of Fig. 1, transition from complete fibrous to complete granular fracture would be less abrupt than for a low-carbon steel. At contact of the curve of flow stress with the ascending portion of the curve of cohesion, the two curves are nearly parallel. Whether sight further upward movement of the curve of flow stress would cause discontinuous movement of the intersection is uncertain. The movement, however, would be rapid. This rapidity of movement, together with the influence of the previously discussed accentuating variable, therefore, would account for rather abrupt transition from fibrous to granular fracture.

As illustrated by diagram *C*, elevation of the curve of flow stress evidently causes no abrupt change in the energy absorption of aluminum. The relationship between the curves of flow stress and cohesion seems to indicate that transition from fibrous to granular fracture of aluminum is gradual. Similar relationship probably would exist between the curves of flow stress and cohesion for most other non-ferrous metals. Such relationship seems to indicate that impact-temperature curves for non-ferrous metals can be lowered by increasing the specific velocity of deformation or the spatial stress ratio, but that the lowering would not be abrupt, as it is for steels.

(14) INFLUENCE OF SIZE OF A NOTCHED SPECIMEN ON TOTAL WORK

The relation between the size of a notched specimen and the total work is of importance not only for comparison of results obtained with test specimens differing in size, but also for application of the results of impact tests in determining the suitability of material for use in machines and structures. One of the earliest hypotheses of the influence of size on total work was that of Kick (10). According to this hypothesis, the total work of similar specimens is proportional to the cube of similar dimensions. The hypothesis, although it is valid for unnotched specimens, has not been found valid for notched specimens.

Attempts were then made by various investigators to relate the total work to the area of cross-section. The International Association for Testing Materials in 1909 came to the conclusion that the total work divided by the minimum area of cross-section is independent of the size of similar specimens and depends only on the metal. This value, known as "specific work," was considered to be a measure of "notch toughness." The International Association for Testing Materials recommended two standard specimens of the Charpy type. The dimensions of the large specimen were 30 by 30 by 160 mm., and of the smaller 10 by 10 by 53 mm. The notch was of the usual Charpy type, extending half way across the specimen and ending in a bored hole. For the large and small specimens, the diameters of the bored holes were 4 and 1.3 mm., respectively. Similar dimensions of the specimens, therefore, were in the ratio of 3 to 1. The distances between supports in use of these specimens were 120 and 40 mm.

Stribeck **(23)**, using these specimens, showed later that the hypothesis of constant specific work with varying size is incorrect. Using a number of different metals, he obtained ratios of total work of the large and small specimens, varying between about 11.3 and 19. According to the hypothesis of constant specific work, the ratio should be 9; according to the proportionality hypothesis, the ratio should be 27. The actual ratio, according to Stribeck's data, is between these values, and tends to increase with the toughness of the metal.

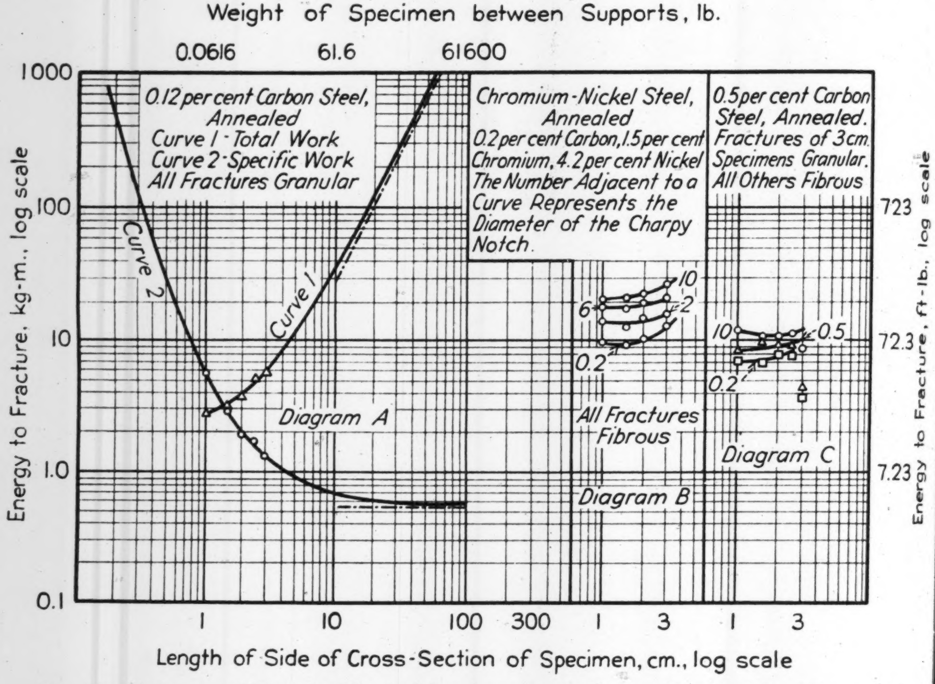

FIG. 13.—Influence of Size of a Notched Specimen on the Energy Required for Fibrous and Granular Fracture.

Moser **(18)**, in accordance with an earlier suggestion by Schüle and Brunner **(20, 21)**, found that the total absorbed energy of steels tends to be proportional to the volume of plastically deformed metal. (This relationship was discussed in connection with Fig. 6.) Except in research, however, this relationship has little practical utility because of the difficulty of accurate determination of the irregular volume of deformed metal. The ratio between total work and volume of deformed metal, moreover, does not always remain constant with change in dimensions of the specimen. With brass specimens of varying dimensions, for example, Bauer, Goeler and Sachs **(2)** found no simple relation between total work and the volume of deformed metal. Nevertheless, the relationship is of considerable theoretical interest.

The effect of size of similar notched specimens and the effect of independent variation of the size of the notch, were investigated by Mailander **(16)**. His specimens were of the Charpy type, of dimensions ranging between those of the large and small standard specimens recommended by the International Association for Testing Materials. The notches extended half way across the section, and the diameters of the bored holes at the bottom ranged from 0.2 to 10 mm. In Mailander's diagrams, ordinates represent specific work (notch toughness) and abscissas represent diameters of notches. Although this method of plotting illustrates important relations, it does not reveal the relationship between either total or specific work and the size of the specimen. Data obtained by Mailander **(16)**, therefore, have been replotted in Fig. 13 to illustrate the relation between absorbed energy and specimen size.

Mailander's investigation was designed to give information about the influence of the dimensions of steel specimens on the total work required for both fibrous and granular fracture. For this purpose, impact tests were made at various temperatures. At 95 C., as shown in diagrams *B* and *C* of Fig. 13, all fractures except those of the 0.5 per cent carbon steel specimens of the largest size were fibrous. At −70 C., as shown in diagram *A*, all fractures (obtained with a 0.2 per cent carbon steel) were granular. Conditions, therefore, were such that this steel exhibited cold brittleness.

When the fracture is fibrous, as illustrated in diagram *B*, specific work evidently tends to increase with specimen size. The slight dip in some of the curves probably does not represent actual decrease in notch toughness with increase in size. In the absence of interfering variables and when the fracture is fibrous, notch toughness probably increases continuously with size, though the change in toughness is slight with increase in size from 1 to 2 cm. The annealed 0.5 per cent carbon steel, as illustrated in diagram *C*, evidently changed little in toughness with increase in size between 1 and 2.5 cm. Further increase in size to 3 cm. was accompanied by abrupt drop in impact value and a change from fibrous to granular fracture.

The evidence represented in diagram *C*, therefore, indicates that increase in the size of a notched specimen may be similar in effect to increase in breadth. As illustrated in Fig. 6, increase in the breadth of a specimen beyond a certain point may cause abrupt decrease in the total work and abrupt change from fibrous to granular fracture. Increase in the breadth of a specimen, as shown in Fig. 5, also moves the steep drop in impact value to higher temperature. It appears logical to conclude, therefore, that increase in size, like increase in breadth, raises the temperature of the transition from fibrous to granular fracture.

Consideration of the influence of the size of a notched specimen on specific work involves consideration of the influence of the size and form of the notch. The effect of the root radius of a notch on specific work depends on the character of the fracture. When the fracture is fibrous, the effect is as illustrated qualitatively by the relative positions of the curves in diagrams *B* and *C*. The smaller the diameter of the notch, the lower is the curve. The quantitative relationship for fibrous fracture is represented in Mailander's paper **(16)** by curves with notch diameters as abscissas. These curves turn downward as they approach zero abscissas, but show no clear evidence of approach to zero notch toughness.

The size of the specimen above which the fracture changes from fibrous to granular probably depends on the sharpness of the notch. With a notch less than 0.2 cm. in diameter, the change of type of fracture might be at a smaller specimen diameter than that indicated for the 0.5 per cent carbon steel in diagram *C*. Whether a notch could be made sharp enough to cause granular fracture (cold brittleness) of the nickel-chromium steel at 95 C. (diagram *B*) is not known. With some metals under some conditions, granular fracture probably cannot be produced by merely increasing the sharpness of a mechanically formed notch.

With cold brittle steel Mailander found no variation in specific work with variation in notch diameter between 0.2 and 10 mm. For granular fracture, therefore, the influence of specimen size on specific work is represented in Fig. 13 by only one curve, curve 2 of diagram *A*. The specific work of cold-brittle steel, as illustrated by curve 2 of diagram *A*, evidently decreases with increase in specimen size, but at a decreasing rate. Increase in size, which has been shown to increase the tendency to cold brittleness, decreases the specific work of cold brittle steel. Because of the practical importance of this influence of size, curve 2 has been prolonged to give a tentative indication of the probable effect of increase in size beyond the limits of actual experimental data. The horizontal asymptote of this curve possibly should be placed even lower than the indicated position. The course of the extrapolated curve, which is based on the only important evidence known to the authors on the influence of size on the specific work of cold brittle steel, indicates that specific work decreases considerably with increase in size beyond 3 cm. For a size of 10 cm. specific work possibly would be only about half as great as for a size of 3 cm. For sizes of 20 cm. or more, notch toughness possibly would be only about one-tenth that of the 1-cm. size. As the diameter of the notch (according to Mailander's data) has practically no influence on the specific work for granular fracture, curve 2 is a general representation of the probable effect of size on the notch toughness of cold brittle steel. This subject, however, needs much additional investigation.

The relationship tentatively represented by curve 2 suggests the probable importance of the relationship between total work and specimen weight. This relationship is illustrated by curve 1. (The indicated asymptote of curve 1, corresponding to the horizontal asymptote of curve 2, slopes at an angle whose tangent is 2. The indicated ordinates as applied to curve 1 represent total work; as applied to curve 2, they represent specific work.) The relation between the total work of cold-brittle steel and the weight of a specimen of Charpy form is indicated by the relation between the ordinates of curve 1 and the scale numbers at the top of the figure. These numbers vary as the cube of the abscissas represented at the bottom of the figure. With increase in specimen size, the ratio of total work to weight obviously decreases rapidly. The distance that a specimen must be dropped to cause fracture, therefore (assuming that it strikes in the correct position), evidently decreases rapidly with increase in specimen size. Assuming tentatively that this relationship is represented approximately by curve 1, the distances of drop required for fracture of specimens 1, 2, 10, 20, and 30 cm. in size would be about 680, 50, 8, 3.7 and 2 ft., respectively. (In these estimates account has been taken of the fact that the mass of a falling specimen, unlike the mass of an impact test hammer, is not all applied at the notch.) The great influence of size on the height of drop necessary for fracture of cold-brittle steel probably accounts for many reported instances of brittle fracture of large structural parts when dropped relatively short distances. In these instances, conditions probably are such as to cause cold-brittleness.

(15) PROBABLE REASONS FOR THE INFLUENCE OF SIZE ON NOTCH TOUGHNESS

The total energy absorbed by a notched specimen includes not only the work of deformation to incipient fracture but also the work during fracture. The work during fracture, which may be a considerable part of the total work, depends very little on the form and size of the original notch, but depends chiefly on the influence of the much sharper, progressing crack. For an understanding of the effect of size on absorbed energy, therefore, consideration must be given not only to the total work, but also to the energy absorbed at various stages of the deformation to complete fracture.

The variation in absorbed energy during impact test has been studied by Schwinnung (22) and others. Variation of force with deformation has been investigated, and results have been represented by curves of variation of force with deformation. These force-deformation curves resemble somewhat the load-deformation curves obtained by slow loading. If the fracture under impact is fibrous, the fracture is not represented by a vertical descent but by a gradually descending curve. If the fracture is granular, fracture is represented by a nearly vertical drop. (The drop is not quite vertical because some energy is absorbed even during granular fracture.) This drop may start from the ascending or the descending portions of the force-deformation curve. A nearly vertical drop following a slow descent may represent a fracture that is initially fibrous but finally granular. The granular portion of the fracture is on the side opposite to the notch. If the surface of fracture consists of alternate fibrous and granular areas, the corresponding graph has alternate stages of sloping and practically vertical descent.

In studying the influence of size on absorbed energy, therefore, it is necessary to distinguish between the work before the beginning of fracture and the work during fracture. The work in each of these stages is represented by the area beneath the corresponding portion of the force-deformation curve. In the absence of force-deformation curves, discussion of the influence of these stages on the form of the curves in Fig. 13 can be only tentative. Attention will be given chiefly to the curves of diagram *A*.

As all the fractures represented in diagram *A* of Fig. 13 were granular, these specimens evidently fractured with little or no plastic deformation in the minimum section. Plastic deformation, however, may occur elsewhere in the specimen even when there is no deformation in the minimum section. This plastic deformation obviously absorbs energy before fracture. The absence of plastic deformation in the minimum section is due to the influence of the high spatial stress ratio; in other sections, the lower spatial stress ratio may permit more plastic deformation. These conclusions are in accordance with reported results of hardness tests, which show that the degree of plastic deformation is greatest at a short distance from the minimum section.

The work during granular fracture probably is negligible in comparison with the work before fracture. The work before granular fracture evidently varies much less than in proportion to the sectional area. Otherwise the specific work would not decrease so much (as indicated by curve 2 of diagram *A*) with increase in the sectional area. With sufficient increase in size, however, the total work evidently becomes more nearly proportional to the sectional area, and the specific work then is little affected by further increase in specimen size. It may be possible thus to account for the descent of curve 2 and its indicated approach to a horizontal asymptote.

(16) USE OF IMPACT TESTS IN SELECTION AND INSPECTION OF MATERIALS

An impact test should not be viewed as a simulation of shock in service. Application of impact testing, moreover, should not be limited to metals that are to be subjected to impact in service. The object of an impact test should be to determine whether a structure or machine, made of the metal under consideration, would be in danger of cold brittleness. The term "cold brittleness," as here used, does not refer to any particular temperature range, but to a condition dependent on the state of stress and on other variables.

In most structures and machines, there are one or more regions of relatively high stress concentration and spatial stress ratio. In these regions, during application or change of load, the specific velocity of deformation is relatively high. The locally high specific velocity and spatial stress ratio elevate the flow stress in relation to the cohesion limit, and thus cause danger of cold brittleness. The danger is increased by anything that tends to move the steep drop in the work-temperature curve to higher temperature, and is decreased by anything that tends to move the steep drop to lower temperature. The greater the size of the structure, the greater is the tendency to cold brittleness, and the less is the specific work of cold-brittle steel.

For a suitable impact test, therefore, the depth and sharpness of the notch, and the velocity of deformation should be more than enough to allow for any effect of form and size of the machine or structure, and also to allow for the possibility that the temperature may be lower in service than during the impact test. If such a test, made on representative specimens, results in the granular fracture and low impact value typical of cold brittleness, the material probably is unsuitable for the intended service. The unsuitability may be due to composition, to heat treatment, or to other causes. If the impact test reveals no evidence of cold brittlenes, there is fair assurance that cold brittleness would not be encountered in the structure or machine. The assurance would be much better, however, if additional impact tests were made at low temperature, especially if the structure is to encounter low temperature in service.

As a measure of ductility and of toughness (with the exception of cold brittleness), much importance should be assigned to the "reduction of area." In the absence of cold brittleness, this property is a good index of the impact value obtainable. In the presence of cold brittleness, the impact value is abnormally low in relation to the reduction of area.

The practical value of impact testing would be greater than it is at present if more attention were paid to the horizontal position and less to the absolute height of the impact-temperature curve. If an impact test reveals no evidence of cold brittleness, only minor importance should be assigned to the numerical impact value. Much effort has been wasted in aiming at specific impact values. As illustrated by Fig. 2, impact values may be highest when the material is in most danger of cold brittleness. The height of the curve of total work, moreover, increases with the velocity of deformation, except within the zone representing cold brittleness (Figs. 3 and 8). In the absence of cold brittleness, therefore, total work is less in slow loading than in an impact test. An impact test that does not reveal cold brittleness, therefore, is less severe than a test with slowly applied load. In the presence of cold brittleness, the converse is true. The quality of the steel, with the exception of its tendency to cold brittleness, is best evaluated in terms of static strength and ductility.

Acknowledgment:

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DISCUSSION

Mr. W. K. Hatt[1] *(presented in written form)*.—The authors have been of service in distinguishing between plastic deformation and resistance to fracture. The ability of a material to transmit "flow stress" at a given velocity is of importance in impact.

The writer, in 1904, presented a paper before this Society[2] in which were reported results from impact loading compared with static loading. It was noted that the velocity of plastic deformation was not very great in the case of steel wires approximately 100 in. in length, corresponding to stretching a length of 2 in. in 0.01 sec. No doubt this velocity was much greater in shorter hard steel specimens. Just what speed of impact stress may be the boundary between normal flow and brittleness under conditions approaching explosive action has not been defined.

Table I.—Comparison of Gradual Loading Tension and Impact Tension Tests.

Gradual on basis of impact tests as 100.

Metal	Diameter, in.	Gage Length, in.	Rupture Work, ft-lb. per cu. in.	Elongation, per cent
Soft steel wire	0.30	72 to 108	96	1.04
Hard steel wire	0.26	108	55	79
Nickel steel	0.50	8	77	98
Soft steel	0.50	8	101	83
Locomotive tire steel	0.62	3	91	33
Average			84	79

Behavior under impact is conditioned by homogeneity of structure and by temperature. Rupture under impact of a non-homogeneous bar developed alternately large and small diameters throughout the length of the bar. Likewise, when a portion of the bar was frozen, a neck developed outside the frozen portion.

A comparison of gradual and impact tension tests, as reported in the paper just cited, is abstracted in the accompanying Table I. It shows on the whole that the elongation under impact was 79 per cent of that under gradual loading and that the work required to produce rupture was in turn 84 per cent.

Table II.—Comparison of Effect of Temperature on Average of All Rods of Soft Steel. Basis of Properties at 70 F. = 100.

Rupture work, 100 = 1550 ft-lb. per cu. in.
Elongation, per cent, 100 = 26.1 per cent
Contraction, per cent, 100 = 64.1 per cent

Temperature, deg. Fahr.	Rupture Work, ft-lb. per cu. in.	Elongation, per cent
−100	38	20
0	62	66
70	100	100
200	102	106
400	76	88

The effect of temperature on these tension impact tests was reported in the case of soft steel and nickel steel as shown in the accompanying Table II from a temperature of −100 F. up to a temperature of 400 F.

The percentages of elongation and work required to rupture in Table II are expressed in relation to these properties at 70 F.

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[2] W. K. Hatt, "Tensile Impact Tests of Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. IV, p. 282 (1904).

Mr. L. B. Tuckerman.[3]—In addition to saying how much I appreciate what I have learned in this discussion, I want to add that one of the powerful tools we have in the study of dynamic problems is dimensional analysis. If we list all the significant physical quantities involved in the dynamic behavior to be studied and combine them into non-dimensional ratios we can predict from the result of one test the result of any other test in which these ratios are all the same. We must remember, however, that we must always put in all the significant quantities. Unfortunately all of our structural metals are polycrystalline, and the size of the crystals is a significant factor in impact tests. The size of the crystals is not changed proportionally to the size of our specimen, so that geometrically similar specimens of different size are not dynamically similar. That is why we do not get anywhere with dimensional analysis in impact tests.

Mr. D. J. McAdam, Jr.[4] (*author's closure*).—In considering the subject matter of this paper, the theoretical explanation given in connection with Fig. 1 should not be overemphasized. Chief emphasis should be given to the relations illustrated by the other figures of the paper. The information given in these figures seems to indicate that results of impact tests should be studied with reference to the entire temperature-impact curve, and especially with reference to the low-temperature drop in impact value.

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SOME CONSIDERATIONS ON THE IMPACT TESTING PROBLEM

By N. Davidenkoff[1]

It has been the practice to design machine parts exposed to impact in the same way as in the case of parts subjected to statical forces. That is, after the momentary stress of the part had been determined by one of the methods appertaining to the theory of elasticity or to the strength of materials, a safety factor in relation to the yield point or tensile strength was properly chosen. Having thus established the working stress, the dimensions of the part concerned is next determined. In making such computations, one is interested only in the magnitude of the safety factor. The author, therefore, has put forward in one of his articles[2] the idea that in such a case there is no reason to differentiate between the two kinds of forces, static and dynamic.

This method leaves out of account the numerical results of the impact test, and the use of the impact test itself is reduced to deciding whether to accept or to reject the material available.

Meanwhile, the idea suggests itself of making a better use of the impact values, which can give a more accurate notion of the danger of brittle fracture, threatening the machine part. This danger can be evaluated by use of a special "factor of safety in relation to brittleness" or briefly, a "brittleness factor." Such a factor must be introduced into our computations complementary to the usual "strength factor" (safety factor).

Let us consider a machine part (for instance a piece of mild steel without sharp corners), which being tested dynamically at various temperatures, displays a well-defined critical temperature of brittleness at which the high impact value is suddenly replaced by a low one, the transition temperature being T_c (absolute). If there exists a zone of scattering, then T_c will denote the upper limit of that zone. This critical temperature of the given material depends evidently on a number of various conditions, namely, the shape of the piece, its dimensions, velocity of impact, etc.

Let the working temperature (also absolute) of the piece be T_o. Then the temperature difference $T_o - T_c$ may be regarded as the distance separating working conditions from those of brittle fracture.

$$\text{The ratio } \xi = \frac{T_o - T_c}{T_o} \quad \ldots\ldots\ldots\ldots \quad (1)$$

can therefore represent the "temperature brittleness factor" or simply the "brittleness factor."[3] If $T_c = T_o$, $\xi = 0$ and there is no guarantee against brittle fracture at all. If $T_c = 0$, $\xi = 1$ and we have the greatest possible assurance of ductility. The intermediate cases are represented by values of ξ between 0 and 1.

If, on the contrary, the transition into the brittle state is gradual (for instance, in the case of a notched bar), the conditions become more complicated. The question arises as to which temperature may in this case be regarded as the critical one. The introduction of two or more new characteristics of the transition zone, for example, the slope of the curve, or its upper and lower limits, is apparently not permissible from the practical point of view.

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[2] N. Davidenkoff, "Allowable Working Stresses Under Impact," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 56, p. 97 (1934).

[3] The author does not by any means insist on this term. "Toughness factor" or "toughness number" may perhaps be more appropriate terms.

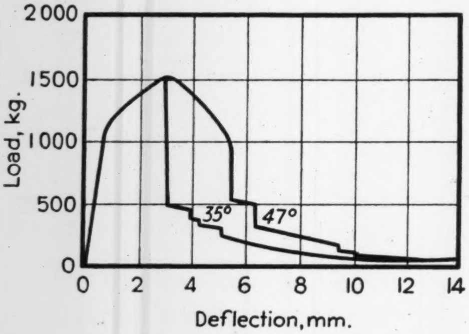

Fig. 1.—Bend Test Load-Deflection Curve for 0.51 per cent Carbon Steel. (Schwinning and Matthaes.)

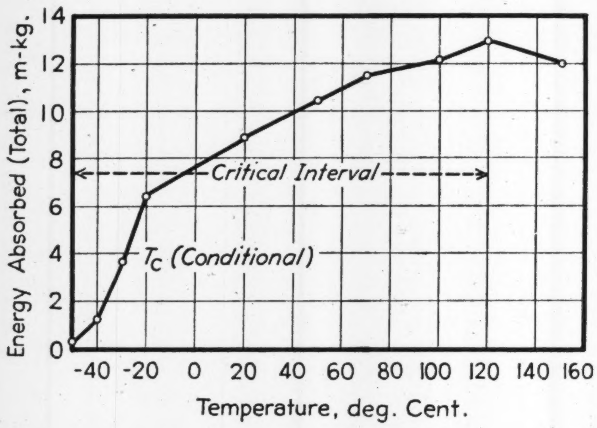

Fig. 2.—Total Energy Absorbed, Charpy Impact Test, for 0.15 per cent Carbon Steel. Notch Fremont type, specimen 10 by 10 by 70 mm.

A suitable method may be adopted, based on some statical experiments which give the "load-deflection" diagrams of notched bars in the transitional zone. There is no reason to believe that a dynamically applied load would change fundamentally the general form of these diagrams (see, for instance the paper by Schwinning and Matthaes).[4] It is well known that at low temperatures these diagrams display abrupt drops, coinciding with the incipience of crystalline fissures on the cross-section of the bar (Fig. 1). The highest point of the curve corresponds to the starting of fibrous fracture.

A comparison between the above test and a standard tension test shows that the maxima on both curves must have the same practical meaning, that is, to characterize the greatest load to be sustained by a given material of a given shape. In choosing the "safety factor" in relation to tensile strength, we pay no attention to the behavior of the material after the maximum of the curve has been attained. Similarly, in the case of bending of a notched bar, the descending branch of the curve with its drops may be regarded as having no practical value. The temperature at which the first drop reaches for the first time the point of maximum becomes the critical temperature, to be used in Eq. 1.

Preliminary experiments show that at

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[4] D. V. M., No. 78 (1927).

the critical temperature the value of absorbed energy falls to about 40 per cent of its initial value (see Tables I and II). On the other hand, experiments often show that the "impact energy-temperature" curve has a point beyond which its slope becomes markedly steeper (towards low temperatures) (Fig. 2); this point also corresponds to an energy loss of nearly the same order (50 per cent). Of course, this value must be verified by more extensive investigations and is here mentioned only as a first approximation in order to give more definitiveness to the following considerations.

TABLE I.—STATICAL BENDING TESTS OF COARSE-GRAINED 0.24 PER CENT CARBON STEEL.
Notched Fremont specimen.

	−140 C.	−110 C.	−100 C.	−40 C.	−30 C.	+40 C.	+60 C.	+80 C.
Maximum load, kg.	460	435	417	577	560	490	495	460
Load of first drop, kg.	460	435	417	577	557	...	...	...
Energy absorbed A (total), m-kg.	0.37	0.37	0.44	2.84	2.90	5.50	5.83	6.54

$$\frac{A_{\text{crit.}}}{A_{\text{max.}}} = \frac{2.90}{6.54} = 0.44$$

TABLE II.—STATICAL BENDING TESTS OF FINE-GRAINED 0.24 PER CENT CARBON STEEL.
Notched Fremont specimen.

	−150 C.	−140 C.	−130 C.	−70 C.	−60 C.	+40 C.	+60 C.	+80 C.
Maximum load, kg.	563	560	655	630	640	545	545	545
Load of first drop, kg.	563	560	655	630	570	....	....	....
Energy absorbed A (total), m-kg.	0.53	0.57	1.12	2.50	2.42	6.38	7.08	6.00

$$\frac{A_{\text{crit}}}{A_{\text{max}}} = \frac{2.42}{7.08} = 0.34$$

It is suggested that if the part is to be subjected to severe conditions, favorable to brittle fracture (impact, low temperature, sharp corners, intensive stress concentration), the constructing engineer responsible for designing the part should be supplied with information not only regarding the strength safety factor, or the working stress, as has been the practice up till now, but also as to the new "brittleness factor." The more important the machine part and the more severe its working conditions, the higher is the value of this factor which should be specified.

The choice of the proper kind of steel can be made by the constructor if there exist methods of evaluating the critical temperature for every material and every variety of condition of its application. How to do this will be stated elsewhere in this article. At present the author wishes to submit some considerations about the general meaning of the brittleness factor ξ.

Investigators have long ago perceived the necessity of replacing individual experiments by a series of experiments, the advantages of temperature as a variable parameter being fully appreciated. However, when an attempt is made to introduce this method into the practice of works laboratories, one often encounters the following objection: Why test the steel at temperatures at which it is hardly ever likely to operate? The answer to this question becomes self-evident after the introduction of the above "brittleness factor."

In fact, it does not seem strange to any one that the ordinary strength factor is calculated in terms of tensile strength, though the working stress in a machine part will never attain this limiting value. Similarly, the determination of critical brittle temperature, though it may never be attained in practice, is necessary for evaluating the temperature brittleness factor. It must further be borne in mind that the temperature parameter is arbitrary and has been chosen as the most convenient variable. In reality, equivalent conditions may be set up by other parameters, for example, by an unexpectedly high velocity of impact or an exceedingly sharp notch, due to an incipient fatigue crack. All these unfavorable changes which can occur in the machine parts in working conditions, result in a rise of critical temperature and thus render the danger of brittle fracture more imminent. The wider the temperature range, which separates the working temperature from the critical one, the more remote is the danger of brittle fracture. Thus the temperature brittleness factor is not at all a factor adopted to guard only against a decrease in the working temperature T_o, which would be unlikely to occur; but is, on the contrary, a factor adopted to guard against an undue increase in the critical temperature T_c of the machine part, which may be produced by unfavorable variations in other working conditions. An occasional fall of working temperature would of course also decrease this factor, so that it must take into consideration the combined influence of all the unfavorable conditions, leading to the convergence of the temperatures T_c and T_o. In other words, the "brittleness factor" is a temperature factor only as regards the method of its calculation, but its physical meaning embraces all possible ways of approximation to the brittle state.

It may reasonably be expected that machine parts having similar T_o and T_c, but made of different materials, will react differently to changes of velocity of impact, the sharpness of a notch, etc., and will therefore be guaranteed from brittle fracture to a varying extent. Unfortunately, investigators have so far failed to reduce the influence of all the factors of brittleness to a single parameter as an independent variable. We must therefore content ourselves with a conditional choice of any one of the factors. Since the temperature factor is the most suitable one, we have singled it out for our purpose. In fact, a change in the form of the notch, apart from the difficulty of expressing it by any one value (radius of curvature? ratio of radius to thickness? depth of notch? the complex function of the contour, according to Kuntze[5]?) is often insufficient to render the specimen brittle. The effect of the variable velocity of the test is not so great as the effect of temperature, since the temperature of brittleness varies approximately in proportion to the logarithm of velocity. The production of brittle fractures by means of only a high velocity of impact involves such velocities as cannot be obtained with any one of the existing impact machines. Temperature therefore remains the only suitable parameter.

Attention is called to a certain defect in the method of evaluation of the brittleness factor as proposed by the author: The absence of any guarantee against fracture (in contrast to the "strength factor") is characterized by the conversion of the factor not into unity but into zero. In the same way the degree of safety is expressed by proper fractions instead of by a number greater than unity. This may be avoided if in place of the ratio shown in Eq. 1, the expression $\frac{T_o}{T_c}$ is chosen; but in this case the coefficient will not be sufficiently expressive. For instance, the fall in the critical temperature from -30 to -60 C., which is in itself very important, would give for ξ, at a working temperature of 20 C., only a slight variation from 1.20 to 1.38, whereas in the previous expression, we obtain values of 0.20 and 0.38, the later being nearly twice as great as the former.

The brittleness factor could also be expressed simply by the difference of temperatures $T_o - T_c$. However it would then lose its nondimensional character, peculiar to all safety factors.

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[5] *Stahlbau*, Vol. 9, p. 121 (1936).

Before introducing the brittleness factor into the calculations of designers, it will be necessary to determine the critical temperatures (or the temperature interval of brittleness) for machine parts of any form and any material, on the basis of data obtained from laboratory tests. Our present knowledge is insufficient for this purpose. Not only do we not know how the transition from the specimen to the machine part is to be made by calculation, but we do not know with any surety all the parameters which will define the critical temperature of brittleness.

It is self-evident that this problem cannot be solved by an infinite number of tests of machine parts of a great variety of shape and size and by the determination *ad hoc* for every shape of its critical temperature. The only correct method would be an experimental and theoretical analysis of the phenomenon with a view to determining those elementary factors which define the critical temperature. Knowing those factors for each machine part and also the kind of material used, it will be possible to determine the critical temperature of the machine part. A successful solution of this problem depends on a proper choice of the factors to be studied. These factors fall in the following categories:

1. Geometrical factors, which determine the shape and size of the machine part.
2. Mechanical factors, which determine the state of the stress.
3. Velocity factor.
4. Technological factors, mechanical and thermal treatment of machine part, and the condition of its surface.
5. Chemical factors, the composition of the steel.

At the same time, the methods of the impact test should also be revised so as to make it possible to obtain, on the basis of experimental results, all the constants necessary to solve the above problem. Such tests will apparently have to be carried out at various temperatures, velocities and with specimens of different shapes.

This important problem can only be solved by the combined efforts of a great number of laboratories. We therefore invite all who are interested in the problem to contribute to its solution and hope that our present appeal will meet with a ready response.

IMPACT TESTS OF WELDED JOINTS—II

A REVIEW OF THE LITERATURE FROM JANUARY 1, 1936, TO JANUARY 1, 1938

BY W. SPRARAGEN[1] AND G. E. CLAUSSEN[1]

Prepared under the auspices of

THE LITERATURE DIVISION OF THE ENGINEERING FOUNDATION—
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NOTCHED-BAR TESTING AND IMPACT TESTING

BY S. L. HOYT[1]

Notched-bar testing and impact testing, though their fields overlap, belong in two separate categories. This is so because the stress conditions produced by a notch and the stress conditions imposed by impact give two different and distinguishable reactions in metals. In this light a test on a notched bar which is broken by impact remains a notched bar test because the notch dominates the behavior of the metal; the impact effect here is of secondary significance and the impact method of determining the amount of energy absorbed in breaking the bar is simply a matter of convenience. A sample of steel which has normal ductility in the standard tension test may break as though it were a brittle material when suitably notched; or, with the same tensile properties, another sample may deform and break like a ductile metal. This variable and unpredictable behavior of steel is due to the fact, we may say, that steel may be "notch sensitive" and the principal object of notched-bar testing is to ascertain the extent to which it is capable of retaining its ductile behavior when notched. By this same token an impact test, if it is to serve the object of ascertaining the reaction of a metal when required to deform suddenly and rapidly, must be carried out on a plain unnotched bar. It has been shown that some steels are particularly prone to break with brittle behavior under impact loading, though they be ductile when broken statically. We may say that such materials are "velocity sensitive" and that the main object of impact testing, though not the sole object, is that of ascertaining the extent to which the materials are velocity sensitive.

In this field of testing it is also necessary to distinguish between metal behavior and mechanics. For example the mechanics of the notch effect are such that even the ductile metals copper, aluminum, etc., can be made to break in a brittle manner if notched sufficiently drastically. In other words we may say that all metals are sensitive to the notch effect, whether loaded statically or by impact. These effects come rightfully in the province of mechanics; it is only when some peculiarity of metal behavior becomes apparent, as in the case of the "notch sensitivity" of steel, that the test becomes one which bears on metal quality or metal behavior. At present it is not always possible to distinguish sharply between these two fields, for neither is adequately understood, but the broad principles may be said to be reasonably clearly perceived. The same situation exists in principle in the field of impact testing. Mechanics teaches that metals respond differently to the impact of a moving body than they do to statically applied loads. In some cases a ductile, yet "velocity sensitive," metal fails in an unpredictable brittle fashion, and the problem becomes one of metal behavior. Again we must distinguish between mechanics and metal behavior.

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One of the main arguments of this discussion is that it is necessary to keep these two distinctions in mind. If we have a clear perception of the property we are determining or the behavior we are studying we ought to be able to set up the test properly and interpret its results intelligently. What is of equal importance, we ought also to be able to diagnose service failures more intelligibly and correlate the findings with the test results. Unhappily the more prominent notched bar test, as carried out here or abroad, leaves considerable to be desired in both ways. I believe the difficulty is principally one of point of view and that with the knowledge now available it is feasible to attempt to set up a more rational procedure for notched bar testing.

THE NOTCH EFFECT

It was observed over a century ago that a notch greatly modifies the stress distribution of a loaded bar and sets up a highly localized concentration of stress at the root of the notch. This opened up a new field of mechanics and in more recent years these nonhomogeneous stress distributions have been studied by means of celluloid models and metal test bars; I believe considerable progress has already been recorded in understanding these stress systems. Fundamentally this is a problem in mechanics. Assuming that the relevant laws are understood, the behavior of most materials when notched could be calculated and predicted with appropriate precision. While I have not carried out the calculations I would expect one could show that glass would be injured by a scratch or a notch in that it would be made to break easily. That this is so for brittle materials is generally known, though I would point out in passing that such behavior is not within the scope of the present discussion. I would also expect one could show that the malleable lead would be benefited by a notch to the extent that it could deform locally to a greater extent than when stretched, for example, in wire form. Under impact loading a notch has the additional effect, by geometrical necessity, of reducing the volume of metal which functions to absorb the energy of the blow and hence to increase the intensity of the stress produced. This notch effect and its influence on the behavior of materials deserves to be emphasized for one is apt erroneously to regard the common notched bar test merely as one which mechanically and empirically solves these problems in mechanics. Of course, one might resort to an actual test bar for such purposes but even that would be the use of the model method for solving a complex problem in mechanics rather than a test of metal quality. The completeness of knowledge of the mechanics of this field has no bearing on the logic of so classifying it; it is still a problem in mechanics.

NOTCH SENSITIVITY

Many years after the discovery of the notch effect and its peculiar stress concentration another discovery was made that brings us directly to the crux of our problem. French engineers found that notched samples of a certain ductile steel broke off sharply when bent in the sense of opening up the notch. The fracture was perfectly brittle and had a crystalline appearance whereas in the tension test the same steel fractured with the usual fibrous character of a ductile steel. Some might ask if all steels would not behave similarly when notched but actually other samples of mild steel with no greater ductility in the tension test were found to deform appreciably in the same notched bar test. This was the particular set of circumstances that led to the notched bar test as we now know it. Thus it was this "unexpected" behavior of steel which was not predictable from the results of the tension test that first inspired the use of a notched bar in mechanical testing and ever since has justified it. I need not dwell here on the inevitability of notch effects in practice but this is complementary to the reason just given for the use of the test. If steels were never subject to a notched condition in service it is obvious that this abnormal behavior would lose technical significance.

This statement of the objective of the notched bar test, in whatever terms it may be couched, not only provides the *raison d'etre* of the test but should also guide its conduct and govern the interpretation of its results. Consequently great significance attaches to the reality of notch sensitivity of ductile steels, and to those who wish to study this behavior I have recommended the use of two mild steels, one of the oxidized type which is used for gas pipe (preferably strain aged) and the other a well-deoxidized type which is used for deep drawing operations. Tension and bend tests fail to distinguish materially between the two, but with a sharp notch, the former is brittle (notch brittle) and the latter is ductile or tough (notch tough). The deoxidized steel can be bent back and forth several times before finally breaking with the characteristic fibrous fracture of a ductile metal. No precise methods of procedure need be resorted to nor do we need a formal test to make the qualitative distinction in notch behavior.

It is perhaps appropriate at this place to note that steel, besides being the classical example, is about the only example of a ductile metal which shows such variable behavior when notched. By this is meant that steel may be now notch brittle and now notch tough though ductile in the plain, un-notched condition. The common face centered cubic metals come in a different category because they do not repeat the behavior of steel. It is true that they too break with a brittle fracture when drastically notched and hence are sensitive to a notch, but they do not vary in behavior as steel does. These non-ferrous metals, or the face centered cubic metals, also remain notch tough at very low temperatures under conditions which produce notch brittle behavior in the steels.

NOTCH BEHAVIOR OF STEEL

Before attempting to discuss tests of the notch behavior of steel we shall next examine notch sensitivity more critically, particularly to see why it is that the notch brittleness of ductile steel is not brought out by tension tests of plain bars. This will be done with the aid of the simple yet brilliant hypothesis of Ludwik which utilizes the conception of the "cohesive strength" of the metal and its relation to the resistance to deformation.

Studies of the plastic deformation of metals have shown that the process is one of slip which is produced by a shear stress. In the tension test the maximum shear stress comes in planes at 45 deg. to the axis of the test bar, and as soon as this shear stress exceeds the resistance of the metal to deformation, plastic yielding occurs. The yield point is that value of the tensile stress whose shear component in a plane at 45 deg. is just capable of overcoming the resistance to deformation. At the same time the load on the normal cross-section produces a tensile stress which is supported by forces of cohesion of the metal. The ultimate value of this strength property of the metal is called the "cohesive strength." A metal that is ductile in tension must necessarily have a sufficiently high cohesive strength to support the tensile load until the shear component produces plastic deformation. (For the moment I am not considering the behavior above the yield point.) Conversely, if the cohesive strength of a metal is less than the yield point stress, the bar must break from the tensile stress before it can yield, and the metal is brittle in tension. This is true whether or not the metal has an operative slip mechanism as has been so clearly demonstrated by tests of single crystals of metals which are ductile or brittle in tension, depending on their orientation. Obviously the cohesive strength of the undeformed metal is a property of great technical interest and Kuntze, for one, has attempted to determine it experimentally.

Ductile extension in the tension test shows that the cohesive strength (of the undeformed metal) lies above the yield point, but there is nothing in the test to show how much in excess of that stress it may be or how nearly the cohesive strength came to being exhausted at the moment deformation commenced. Thus the tension test of a ductile metal provides only a lower limit for the cohesive strength, below which we know it cannot lie. Returning to the notch-tough and the notch-brittle steels which were equally strong and equally ductile in the tension test, their ductility shows that in both cases the cohesive strength was high enough to support tensile elongation. Aside from that the cohesive strengths of these two steels might be vastly different and we might find a marked difference in behavior with a stress system which intensifies the normal or tensile stress without affecting the shear stress.

The application of simple mechanics to the tension test shows that the intensity of the tensile stress is just twice that of the shear stress in the planes at 45 deg. to the axis. In further precision of our understanding of a metal that is ductile in tension, we may say that it is one whose cohesive strength is at least twice its shear strength or resistance to deformation. The extent to which the ductile behavior can be altered by a notch becomes clear from a closer inspection of the notch effect. Figure 1 shows an ordinary tension test specimen at the left and a notched bar at the right. It can be seen that the areas of the planes at 45 deg. (which pass through the cross-section at the base of the notch in the notched bar) have not been changed by the notch. The minimum area supporting the tensile load has been reduced by the notch so that, other

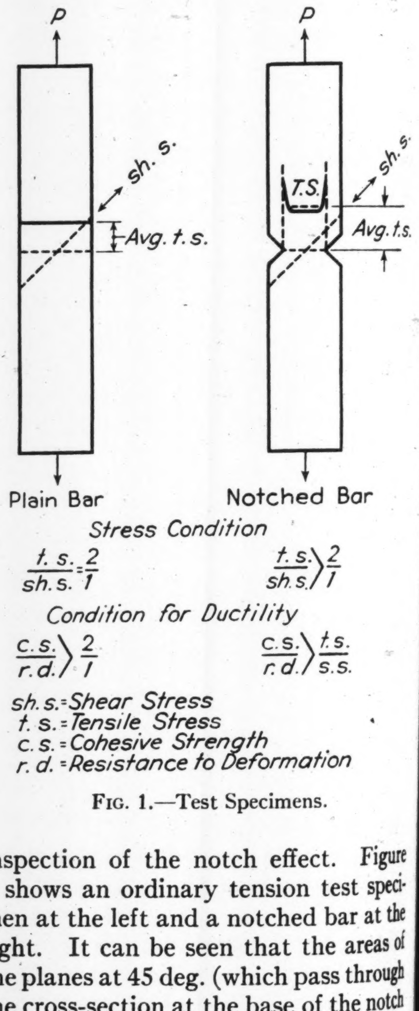
P
P
sh. s.
Avg. t. s.
T.S.
sh. s.
Avg. t.s.
Plain Bar
Notched Bar
Stress Condition
$\frac{t.\,s.}{sh.\,s.} = \frac{2}{1}$
$\frac{t.\,s.}{sh.\,s.} > \frac{2}{1}$
Condition for Ductility
$\frac{c.\,s.}{r.\,d.} > \frac{2}{1}$
$\frac{c.\,s.}{r.\,d.} > \frac{t.\,s.}{s.\,s.}$
sh. s. = Shear Stress
t. s. = Tensile Stress
c. s. = Cohesive Strength
r. d. = Resistance to Deformation

FIG. 1.—Test Specimens.

things being equal, the load required to produce deformation along the 45-deg. planes would require a correspondingly higher tensile stress at the cross-section of the notch. This means that the ratio of tensile to shear stresses must be greater than 2:1 in the notched bar. As a second feature of the notch effect, the tensile or normal stress at the root of the notch is greater than the average over the cross-section. This is indicated by the curved stress line which brings out the marked stress concentration at the notch. In other words, in addition to the higher average tensile stress, in the notched condition the stress at the very root of the notch mounts to some still higher value which varies with the sharpness of the notch. Finally the metal outside the notch adjacent to its walls carries a relatively small load. This metal opposes the natural constriction of the bar and sets up a condition of triple tensile stress in the notch area.

These three factors contribute to an increase in the tensile stress which is required to produce plastic deformation along the planes at 45 deg. As compared to the plain bar, the notched test specimen subjects the metal to a radically different stress distribution in which the ratio of normal to shear stresses rises above the ratio of 2:1 of the tension test. The actual value of the ratio depends on the geometry of the bar and the stress concentration factor. In the case of the notch-tough steel in the simple test previously reported, the cohesive strength was still capable of supporting the normal stress up to the time plastic deformation started and hence conditions for ductile behavior were satisfied. The cohesive strength of the notch-brittle steel was so low that, with the same notch, the normal or tension stress produced brittle cleavage before the shear component of the applied stress overcame the resistance to deformation. We might also say that the notch effect was sufficiently drastic to search out the low cohesive strength of one steel but was still inadequate to do so with the other. Another notch with a higher stress concentration factor might produce a normal stress above the cohesive strengths of both steels and under such conditions both would be brittle. It is obvious that the cohesive strength is a controlling property and that the possibilities of ductile deformation depend on the ratio of that property to the resistance to deformation. The strong heat-treated steels have a very high cohesive strength but the resistance to deformation is also high so that such steels have generally lower ductility and notch toughness.

If one knew the two controlling properties, cohesive strength and resistance to deformation, and if he thoroughly understood the laws of the notch effect, he would be able to predict by calculation whether a given steel would deform when notched, or open up a crack. Unfortunately we do not commonly determine the cohesive strength independently and hence this field cannot yet be treated mathematically. For the present we must continue to use an empirical test and determine from the notch behavior under arbitrarily fixed conditions what we need to know about the cohesive strength. Such a procedure is logical enough and might take the following lines, in principle.

In practice we are usually dealing with a steel that is required to have a certain set of mechanical properties, which means a certain fixed yield point or resistance to deformation. Our problem becomes that of ascertaining whether its cohesive strength is adequate for the use to which that steel is to be put. This can be done by means of what I should like to call the "matching" principle, though it will be shown later that advantage can be taken of a materially abbreviated procedure for testing purposes.

In the discussion of the tension test we learned that the steel is ductile only if the cohesive strength is more than twice the resistance to deformation. In a notched-bar test the requirement for ductility is stiffened somewhat in that the lower limit of the cohesive strength must be still higher relative to the resistance to deformation. Let us assume a 3:1 ratio of tensile stress to shear stress for a given notch. That means that a steel, to retain its ductile behavior, when notched, must have a cohesive strength that is at least three times the resistance to deformation. If the ratio of the two strength properties is only 2.5:1, the steel is ductile in the tension test but is brittle when subjected to that notch effect. In other words it appears that the behavior of a metal when notched depends (1) on the ratio of the tensile stress to the shear stress which is established by the notch, and (2) on the ratio of cohesive strength to resistance to deformation of the steel. Only in case the strength ratio exceeds the stress ratio is the steel ductile. In a series of tests in which the notch effect increases in severity, or in which the ratio of the two stresses increases from bar to bar, at that bar for which the stress ratio "matches" the strength ratio, the steel will change in behavior from ductile to brittle. By such an empirical procedure we could establish the level or magnitude of the cohesive strength, in relation to the resistance to deformation, or we could compare the cohesive strengths of two steels which have the same resistance to deformation. In general we could locate the level of the ratio of cohesive strength to resistance to deformation, or the degree of notch sensitivity, by this "matching" principle. It is not suggested here that such a series of test bars is necessary for testing purposes but we see that it is necessary to find the notch condition which makes the steel break brittle before we can establish the actual level of the cohesive strength.

I have taken the privilege of simplifying this discussion of the notch effect by confining my remarks so far to the conditions prevailing up to the moment plastic deformation begins. What happens after that relates quantitatively to the amount of deformation but does not affect the character of the initial response to stressing. It may happen that the conditions which lead to failure, or to crack formation, do not become established until after some amount of deformation or bending has occurred. Kuntze has shown by his tests that both cohesive strength and resistance to deformation are affected by plastic deformation and each in its own individual way. Judging from his curves the cohesive strength increases at first, then passes through a maximum, and finally decreases. The resistance to deformation increases throughout the course of deformation and, even at the start, at a somewhat faster rate than the cohesive strength. Let us suppose that a notched bar is initially ductile; after deformation proceeds the two controlling properties change accordingly, and as soon as the resistance to deformation "overtakes" the cohesive strength, in effect, the conditions for crack formation become established and the bar fractures. At the same time, however, this same deformation also reduces the stress concentration by rounding out the curvature at the bottom so that the stress conditions, as well as the strength properties, are also altered by any plastic deformation. The actual conditions over this stage become quite complex and a running analysis would be required to give the true behavior. Much more work is needed here before we can satisfactorily picture the notch conditions after deformation has commenced.

VARIABLES OF NOTCHED-BAR TESTING

The history of notched-bar testing shows that considerable attention has been paid to the design of the test bar. Investigators found the notch behavior of a material to vary greatly with the size and geometry of the test bar, and particularly with the notch. It was especially disconcerting to find that those variations themselves changed from material to material. We may list these variables as (1) the size of the bar, though otherwise geometrically similar, (2) the size of the bar, with the same notch, (3) the width of the bar, (4) the height or depth of the bar, (5) the depth of metal broken, (6) the depth of the notch, (7) the angle of the notch, and (8) the radius of the notch or its sharpness. It was found, among other things, that the depth of the notch, the angle of the notch, and the width of the bar had a smaller effect on the tougher steels than on the more brittle type. We can see now that the variations which were noted actually brought out characteristics of the steels tested, and I believe it is logical to use these variables in tests of notch sensitivity. Before passing on I wish to point out that a study of these variables shows the necessity for using a notch of sufficient depth and intensity to avoid spreading the deformation to the outer surface. The latter condition introduces the elements of the simple bend test which, by permitting the metal at the notch to deform, changes both the properties of the metal and the stress conditions of the notch. This increases the energy absorption and destroys the use of such a test as a notched-bar test.

ENERGY OF DEFORMATION

No mention has been made as yet of the amount of work done in breaking the notched bar, though it has a certain obvious use for evaluating the notch toughness of steels and is actually so used in present tests. The Charpy test with its 5-mm. deep notch and the Izod test with its sharp V notch rely almost solely on the impact value for judgment of the steel. I have indicated that the criterion by which the notch sensitivity is to be gaged is given by the "matched" conditions required to produce brittle failure. It seems pertinent, therefore, to raise the query if the impact value can make adequate distinction between tough and brittle steels and, if not, what use may legitimately be made of the impact value.

The energy absorbed in breaking a notched bar—the impact value—summates the work done in deforming the metal at the notch and the work done in cleaving the two faces. It has been shown that the latter component is relatively small and may usually be neglected. This means that the impact value gives a measure of the amount of deformation and hence the reaction of the metal being tested to the particular notch effect used. In other words a low impact value shows that the bar broke with a brittle fracture and a high impact value shows that considerable deformation occurred. On this account the impact value can be used to judge the ductile or brittle behavior of the individual bar though certain limitations control the use of the impact value as a measure of notch toughness.

The impact value makes no distinction between high strength combined with low ductility and low strength combined with high ductility. A strong steel, or one with a high resistance to deformation, can have the same impact value as a weaker steel with greater ductility, yet common experience teaches that two such steels may differ greatly in notch behavior. In about the same way, a soft annealed steel and a harder heat-treated steel may have the same impact value, let us assume 8 ft-lb. It does not follow that these steels should be rated equal in notch behavior; the former might receive a low and the latter a high rating. The former has a low cohesive strength for its resistance to deformation and the latter has a high resistance to deformation for its cohesive strength. Obviously the impact value cannot be used as the sole criterion of behavior, and in practice this is recognized by placing individual requirements on the various steels and treatments.

I have already noted that the impact value summates the work of deformation throughout the entire course of bending and fracturing the bar. This includes the energy absorbed up to the moment of crack formation as well as the energy absorbed in tearing the two halves apart. From information available it would be useful to determine separately the amount of energy expended and the corresponding amount of deformation up to the point of crack formation. This crack might form in one steel as soon as deformation commenced or at a later stage in another, yet though the total energy absorption were the same our judgment of the service reliability of two such steels might be quite different. The stress-strain relationships have been determined experimentally for both impact and static loading. The latter type of test makes it readily possible to determine the point of crack formation, to give a much more adequate understanding of the nature of the material being tested than can be obtained from the energy of rupture alone.

TEMPERATURE SENSITIVITY

Temperature is known to have a marked effect on the notch behavior of steel, much more so than it does on the tensile properties. Whereas the tensile strength and tensile ductility change gradually and slowly over the range of atmospheric temperatures, the notch behavior of the same steels may change abruptly at some temperature within that range. The actual temperature at which the change comes is a function of both the notch sensitivity of the steel and of the notch used in the test—the more severe the notch and the more sensitive the steel, the higher the temperature. With the keyhole and V notches this change occurs in many steels at about room temperature or between room temperature and the freezing point, while in others the change does not come until the temperature has fallen to well below the freezing point. One corollary of this is that a high impact value at the ordinary test temperature is no insurance of ductile behavior at a lower temperature, with the same notch conditions. Another is that at low temperatures the various types or qualities of steel all behave in a brittle manner, and at high temperature (that is, above room temperature) they all become relatively ductile. This holds, of course, for the notches commonly used in testing. Coarse grain size, mechanical hardening, certain embrittling elements, an "oxidized" condition, etc., tend to raise the temperature of the change point, while ductilizing and refining heat treatments, fine grain size, the addition of certain alloying elements, and a "deoxidized" condition tend to extend notch toughness to lower temperatures.

The use of the matching principle for the detection of notch sensitivity may be said to hold for one temperature. The same comments might be repeated for any other temperature; the problem at the new temperature would remain essentially the same. In other words, changing the temperature alters the fundamental properties which govern notch behavior and the problem is simply shifted to the new temperature. By its effect in changing the behavior of a steel from ductile to brittle with a fixed notch effect, lowering the temperature repeats the effect of intensifying the notch effect at constant temperature. In the first case the fundamental properties change to "match" the particular stress conditions of the test bar used, and brittle behavior ensues. In the latter case, intensifying the notch effect does not alter the fundamental properties, because the temperature remains the same, and the "match" is secured by altering the stress conditions of the test bar. Thus temperature is an entirely different type of variable though superficially it appears to have the same effect as changing the notch. To bring this into line with other facts of notch behavior, Maurer and Mailänder assumed that both cohesive strength and resistance to deformation increase with drop in temperature, though with the cohesive strength being affected less rapidly than the resistance to deformation. For a given notch a temperature is ultimately reached at which the fundamental properties "match" the stress conditions of the notch used and the rapid drop in impact value is recorded. This temperature depends on the notch used as well as on the properties of the steel being tested.

The point that is most significant to notched-bar testing is the necessity of making use of test temperatures below the usual laboratory temperature if the steel is to be used at lower temperatures. It would also appear to be sound procedure to use a test temperature at which, for the steel being tested, the maximum spread in impact values is observed for the various conditions of that steel. That would doubtless have considerable practical utility but on strictly theoretical grounds the use of a low test temperature is required only in case the steel is to be used at a low temperature. A good test at room temperature gives no assurance that the steel will remain tough at the service temperature and may very improperly give an assurance of adequacy when such does not exist in fact. By the same token the test temperature ought not be much below the service temperature, for a satisfactory grade of steel would be inadvertently condemned along with an unsatisfactory grade.

VELOCITY SENSITIVITY

Notched-bar tests are made in practice on a pendulum type of testing machine, for it was early found that the energy required to rupture the bar is most conveniently and accurately made in that way. At the same time it was realized that the actual striking velocity might itself affect the results. Tests on this point showed it to be relatively unimportant over the range of striking velocities used. When static tests were compared to impact tests it was found that tests bars of the tougher steels absorbed more energy as the speed of deformation increased. The difference might be of the order of 25 per cent, or even as high as 50 per cent in some cases, and was undoubtedly due to frictional energy that had to be supplied when the metal was deformed rapidly. This energy appeared as sensible heat and is presumably of the same source as the energy differential found when plain unnotched bars are broken statically and by impact.

It is generally held that the stress required to produce a given deformation increases with the speed of deformation and there is some evidence that the increment of stress can be very large. This acts automatically to increase the normal or tensile stress and may be expected first to increase the energy of deformation and then to produce brittle failure. From the results available the change from ductile to brittle behavior must be very uncommon for the velocities used in testing. At much higher velocities, Hadfield and Main have shown that some steels become brittle at bullet velocities, though they were of the overheated, coarse-grained type. A velocity effect is more commonly found when the variables of notched-bar testing are studied "in combination," with one of the variables being the speed of deformation. Thus when notched bars are broken statically they remain ductile at lower temperatures, in greater widths, and with more severe notch effects, than when broken dynamically. In other words, if the notch effect and temperature are critical, the change or "match" between the stress conditions and the strength properties is established and a change from ductile to brittle behavior is produced by increasing the speed of deformation. In actual testing it appears that only under such critical conditions are steels "velocity sensitive." Under service conditions, with higher deformation velocities, materials must be more apt to develop the brittle behavior. Chromium steels may be an exception to the general rule for they have been reported to be affected by relatively low velocities. I have observed a case recently which I believe to be explained largely on the basis of velocity sensitivity which was due to the conditions being critical. The ordinary impact values of this steel were satisfactory but in service a rather sharp crack was present, unknown until later, and the temperature was about 25 deg. Fahr. below the test temperature. Under a sharp impact, the crack spread into a sudden fracture through the entire section.

THE NOTCH EFFECT IN PRACTICE

It is proposed here that tests for notch sensitivity must take into account both the characteristics of notch behavior and the requirements imposed on a part by service, if such testing is to fulfill its proper function in the selection and treatment of steel. The former guides the details of the test used and the latter controls the requirements for acceptance. Furthermore, experience with the steel, both in tests and in service, is essential to the proper conduct of the tests and the interpretation of the results. Of course this reflects the essentially technological or empirical character of the test, though it must also be ascribed in part to lack of engineering data. So important is this that it will be desirable to consider notch effects met with in practice.

It has been pointed out that notched-bar testing is important because the notched condition is actually encountered in practice. Such notches in milder form may be oil grooves, fillets, various changes in cross-section, holes, splines, keyways, gear teeth, bolt and rivet heads, etc. Modern engineering design lessens the dangers of these features by providing generous transitions and producing as nearly streamline flow of stress through the part as possible. Even so a mistake here, or an error in production or assembly, can undo good engineering design. Notches of a more critical nature are screw threads, junctions in assembly, and other transitions in section of an abrupt character. These "external" notches are obvious to the eye and hence are usually held under control.

We have another group of "internal" notches which are less subject to control because not usually visible. Examples of these are flakes in forgings, bursts, heat checks, grinding cracks, shrinkage cracks in castings and welds, transverse fissures in rails, quenching or hardening cracks, coarse slag stringers, coarse phosphide bands and segregations of a brittle constituent. To these may be added the fatigue crack which forms a very effective notch. I have just recently examined one such which was about 1½ in. deep and 6 in. long that led to the sudden failure of a steel block over 30 by 60 in. in cross-section. Coarse machine marks, particularly when on fillets, stamped numbers, etc., are dangerous, for they may produce a fatigue crack under alternating loads which later causes sudden failure. A carburized surface is likewise a hazard for it has little resistance to crack formation and hence may readily produce a serious crack for the metal underneath. Corrosion pits and corrosion cracks may also produce a serious notch effect. Hairline cracks are particularly prone to form in the surface of certain alloy steel forgings. This occurrence has been given by one metallurgist (Greaves) as a reason for avoiding the temper brittle condition in such steels. Finally the presence of internal stresses must, in certain cases, act in the same manner as a notch. The notch effect is resolvable into a stress effect in the metal, and if internal stresses occur of the correct sign and in the correct location, the application of an external load must produce the same result as though that stress condition had been caused by a notch.

These notches, external and internal, intentional and adventitious, are the hazards to which parts in service may succumb from the workings of the notch effect, and to which the more notch-sensitive type of steel is particularly susceptible. They vary considerably in shape, angle, depth, and in radius of curvature at the bottom. These are all variables which affect the normal or tensile stress, the concentration of stress at the root, and the extent to which the condition of triple tensile stress is established. Under most conditions they ought to be avoided but the chief object of mentioning them here is to show first how impossible that is to accomplish, and second how difficult it is to plan a rational test to take them properly into account for any given application.

CAUSES OF LOW NOTCH TOUGHNESS

On the average, steels of low notch toughness are more apt to fail than steels of high notch toughness, or, with specified tensile properties, a steel is more apt to fail the lower its cohesive strength. The causes which appear to be particularly effective in increasing the notch sensitivity of steel are as follows:

1. Casting structure.
2. Overheated forging structure.
3. Overheated structure from heat treatment.
4. Temper brittleness.
5. Low temperature.
6. Strain age embrittlement.
7. Cold work.
8. Certain alloy compositions.
9. High resistance to deformation.
10. Embrittling elements, such as phosphorus, silicon, particularly in high-carbon steels.
11. Coarse segregations and laminations.
12. "Condition" of steel as affected by melting, refining, and deoxidation practice.

These conditions may occur singly or in various combinations, and may or may not be detectable by microscopic examination. Their effects have been brought out by standard notched-bar tests, either by a low impact value or by the temperature corresponding to the change from high to low impact values.

TESTING NOTCH SENSITIVITY

In the present test, as commonly made, one or more bars are broken at room temperature and the impact value obtained. The impact value is taken as a measure of the notch toughness but inasmuch as different steels have different and arbitrarily assigned standards, the impact value is actually used more as a rating, which is proper. When intelligently used, by which I mean when supported by a broad experience with the steel in the application for which it is to be used, this simple test has demonstrated its value and utility for forty years. Even so, it is an incomplete test of notch sensitivity and in some cases can give misleading results. Furthermore, the developments of recent years in the theory of notch sensitivity make it practical to set up a more rational testing procedure.

Whether the notch is the Izod V notch or a Charpy notch which is 2, 3, or 5 mm. deep, the test shows no more than the behavior with that particular notch. We may say that a standard test "samples" the behavior at a fixed point but does not show how the steel would react to a different notch effect. A complete procedure would require the use of a graded series of test bars which would give the position of the "match." It would be impractical to determine this point each time but for more important and severe applications it is highly desirable to secure evidence at one or two additional points. I have recommended such a test in the past which is an outgrowth of the work of Moser on bars of variable widths. Moser used the large Charpy bar (30 by 30 mm.) and compared the behavior of bars ranging from ⅓ to full width. Experience has shown that it is more practical to use the 10-mm. bar as the standard, and this makes it possible to compare bars of single, double and even triple width. The single width bar gives the standard impact value. If the steel retains its same (ductile) behavior at double (or triple) the standard width, the impact values follow closely the same relationship. It happens sometimes that the "match" point falls in between two of the bars and this is shown by a drastic fall in the impact value, as compared to the extrapolated value. A typical case of this kind is illustrated in Table I. The data were kindly supplied by Mr. G. H. Wright of the General Electric Co. The test results represent the same steel in two different conditions of heat treatment. The tension test and the standard Charpy test show equivalent behavior and the two conditions here represented would have to be given equal ratings. After breaking the double width bars, the two could no longer be regarded as equivalents and the first condition would be seriously questioned, or actually rejected (or re-heat treated), for the more severe applications. The making of a double width bar is simple enough to justify its use whenever the occasion warrants.

TABLE I.—SINGLE-WIDTH *versus* DOUBLE-WIDTH BARS.

	Elastic Limit, lb. per sq. in.	Tensile Strength, lb. per sq. in.	Elongation in 2 in., per cent	Reduction of Area, per cent	Impact, ft-lb.	
					Single-Width Bar	Double-Width Bar
Condition 1.....	55 000	91 400	28.5	61.8	19.5	8
Condition 2.....	58 000	96 000	26.5	60.0	19.0	37

In principle a similar test could be devised with 10-mm. square bars but with notches of varying intensity. The notch effect has not been closely enough analyzed as yet to set up as satisfactory a test on this basis. The mathematical relationship between the single and double width bars makes it very simple to compare the actual double width value with twice the single width value to secure the judgment of the steel.

Temperature has a prominent effect on notch toughness, yet this is not ordinarily taken into account in testing. The only exception to this is the tests which are made at –50 F. or lower for steels which are to operate at such low temperatures. This leaves a gap in testing practice for those parts which must operate outdoors or at intermediate temperatures. The standard test is made at room temperature and ignores the circumstance that the same notch might produce brittle failure and a low impact value at a temperature but slightly lower. On this account a lower test temperature may be required to indicate the probable behavior in service; more severe tests at room temperature or the requirement of a higher impact value do not offer a substitute because lowering the temperature actually changes the controlling properties.

Sub-normal test temperatures are also well suited to testing steel in general, for there is a certain parallelism between the steel quality that tends towards notch sensitivity at room temperature and the steel quality that loses notch toughness at a relatively high temperature. Hence if a steel retains about its room temperature impact value when cooled down to the freezing point, it may properly receive a good rating. Thus temperature has utility in notched-bar testing far beyond the use which is now made of that variable. In judging impact values for these purposes it is necessary to know the "low level" values and to recognize the change from the high to the low level. If the notch produces the low level values, lowering the temperature has but a small effect.

Standard notched-bar testing leaves out of account two additional features which have an important bearing on the judgment of steel. These have already been mentioned but may be repeated here, namely, the stress-strain relationships and the moment of crack formation. These can be conveniently determined in a static test, and if the energy value agrees properly with the standard impact value we can appraise the two factors of strength and ductility which comprise the impact value. At the same time the moment of crack formation can be ascertained and the amount of energy absorbed up to that point determined. A steel which is capable of deforming, though under constraint, without forming a crack, possesses a valuable characteristic for some service. We may also say that a steel which resists the spreading of a crack, once one has formed, possesses another valuable characteristic. By means of a static test, the amount of energy absorption and the deformation for both periods can be secured. Interesting results of this kind have already been obtained and are capable of providing valuable information with which to judge the different steels and their treatments.

It has been mentioned that the convenient method of determining the work done in rupturing a notched bar is by the use of a machine of the impact type. Various questions have been raised as to the accuracy of this method, particularly because energy can be lost by vibrations in the machine and by scraping the bent test bar along the sides of the split anvil. In so far as I know, it has never been asserted that the change from ductile to brittle behavior is missed by errors in the test method. On the other hand, it has been shown that good check results are obtained with duplicate test bars from a homogeneous bar of steel. These points have been fully treated in the literature and need not be discussed here at length. Of more recent date, results of the "Oxford" machine have become available. The energy required to break the test bar is obtained practically without experimental error, yet the Izod values of companion bars agree so closely with the "Oxford" values that the rating of the steels would remain the same. When we take into account the character of the determination being made it is clear that the present type of impact testing machine is well suited to the task.

IMPACT TESTING

Upon turning to impact testing, I believe it desirable to point out that the notched-bar test which we have been discussing is commonly called "the impact test." An analysis of notch behavior shows this to be an incorrect use of the term, which results in considerable confusion of ideas, and for a better definition we may say that an impact test is one which has as its object the determination of the behavior of materials under impact loading. As was shown, this excludes the notched type of test bar. We may also exclude from consideration such tests as "drop tests" which are used for railway materials, etc. While it is frequently convenient to use an impact to apply a load, such tests are not designed primarily to test the mode of reaction of the material to a rapidly applied stress or impact.

We have again to distinguish between mechanics and metal behavior. Impacts set up stresses in the absorbing body, and if rupture is to be avoided the energy of the blow must be taken up by elastic and plastic deformations. It is well known that the stress generated increases as the volume of metal affected decreases, while brittle metal is particularly susceptible to failure by shock because it is incapable of absorbing the blow by deforming plastically. While metallurgical problems are involved, these matters are principally the concern of mechanics and need not be considered here. From that point of view we do not need an impact test except to determine the quantitative difference between static and dynamic loading. The principal concern of an impact test of metal quality must be the detection of abnormal behavior, such as a change from ductile to brittle fracture of "velocity sensitive" materials. The latter comes within the province of metallurgy and offers a sound reason for testing metal quality under impact loading.

Examples of velocity sensitivity, apart from those cases cited under notched-bar testing, are extremely rare if we can judge from the frequency with which this subject is treated in the technical literature. I know of no more simple case than that reported at an early date by Considère. His results were published many years ago and have been discussed on various occasions since then, so only the principal findings need be covered here. The steel he used was very likely above the average in velocity sensitivity and serves well to illustrate the effect. Considère tested small (unnotched) bars by means of weights which fell from various heights to break the bars at various striking velocities. In each case he determined the energy absorbed in fracturing the bar and noted the plastic behavior. Below a certain velocity (height of fall) the steel deformed in a ductile manner, as it did in the tension test, and the energy absorbed was commensurate with the deformation. Above this "threshold velocity," the steel broke in a brittle manner and the energy absorbed fell off simultaneously to a correspondingly low level. This gives a classical example of "velocity sensitivity" of a ductile metal and the term implies that the metal becomes brittle in impact above a fixed striking velocity or speed of deformation. Possibly all metals would change in this same way if we could arrange to deform them fast enough, but many of the ductile metals retain their ductile behavior at least up to bullet velocities of the order of 2000 ft. per sec. As an exceptional case Hadfield and Main found that a normally ductile steel in an overheated condition became brittle at about 1700 ft. per sec. Except for known cases of velocity sensitivity, we may say that metals retain their ductile behavior and toughness when deformed by impact, though in this field of testing we know too little about the behavior of the various metals and the causes of velocity sensitivity. I have personally observed that a steel which ran 8 ft-lb. in the Charpy test was perfectly ductile to the penetration of a bullet.

Temperature is commonly held to have a profound effect on the impact behavior of metals, and particularly of steel. This was also studied by Considère and he found that while temperature had practically no primary effect on either the ductile behavior or on the energy value, it had the very important secondary effect of shifting the threshold velocity to lower values as the temperature was lowered. Apparently the temperature at which steel changes from ductile to brittle, for a fixed striking velocity, depends on the structure and possibly on other metallurgical characteristics. Davidenkoff found that an overheated boiler plate became brittle (under his test conditions) at −90 C. though the same steel in good condition remained ductile down to −150 C. On the other hand, the temperature at which a change from ductile to brittle behavior takes place depends on the manner of stressing or straining the metal, as Heindlhofer has made so clear. It is not correct to say that a metal becomes brittle at some low temperature if we mean that ductility is a property or characteristic which the metal loses at that temperature, or that the metal has an operative slip mechanism above but not below that temperature. Ductility is not an independent property but, in metals which are capable of plastic deformation, is a manifestation of plasticity which occurs when the shear stresses which produce deformation are not accompanied by tensile stresses which exceed the cohesive strength. Hence the type of behavior depends on the particular stress system employed. Thus Heindlhofer showed that the same steel "became brittle" at different temperatures when tested in torsion, tension, and by a notched-bar test. The properties of the steel are fixed at a given temperature and the difference was due to the stress systems employed.

I have referred above to a small quantitative difference in energy absorption between static loading and impact loading at striking velocities below the threshold velocity. Precise determinations have shown that metals usually absorb more energy when broken by impact than when broken statically. This increment is frequently of the order of 25 per cent but may be 50 per cent or more. On the other hand it is also reported that some metals break with a lower energy absorption when broken by impact, examples of which are the 18 per cent chromium, 8 per cent nickel stainless steel and possibly the chromium steels. The actual strength is greater, as shown by the stress carried by the test bar, but the elongation is enough lower to account for the smaller energy value. A case of this kind is reported by Clark and Dätwyler in this symposium[2] and can be accounted for by the effect of the rate of loading on the true stress as has been done by Kuntze and by McAdam and Clyne.

By way of summarizing the impact

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[2] D. S. Clark and G. Dätwyler, "Stress-Strain Relations Under Tension Impact Loading," see p. 98.

behavior of metals, it appears that they generally repeat their static behavior, with due allowance for the effect which the laws of mechanics may have on the impact behavior. Some steels develop brittleness under impact at lower striking velocities and at higher temperatures than do other steels. The change from ductile to brittle behavior is abrupt and is coincident with a sharp drop in impact value. This is consistent with the view expressed above that plastic deformation absorbs energy, or requires work to be done, but that cleavage absorbs but little energy. There appears to be no good reason to believe that metals change materially in behavior at room temperature as long as the velocity is below bullet velocities, unless the metal is velocity sensitive.

More recently Mann, and Haskell and Mann, have reported results of high-velocity impact tests, made with a special machine, which present quite a different picture of impact behavior. This work is well known and I believe we need note only the points of divergence. The energy absorption is reported to be constant over the lower range of striking velocities, though the velocities used are high enough to bring out the quantitative velocity effect which has been reported by other investigators. Above a certain velocity, called the "transition velocity," the impact value decreases slowly and gradually with further increase in striking velocity, instead of falling abruptly from a high level to a low level which it would do if this were the threshold velocity. Over this range of falling impact values, the elongation and reduction of area are reported to increase, instead of decrease as one would expect. Unfortunately no values of the stress have been reported. In one case the striking velocity was carried up to about 200 ft. per sec. and the test bar was reported to elongate by its full amount but with no measurable energy absorption. Obviously this would mean that the metal was deformed and broken without setting up any resisting stress and without requiring any work to be done on it. The behavior reported for this range of striking velocities does not conform to the laws of plastic deformation and needs an explanation. On the other hand the change from ductile to brittle behavior at the threshold velocity was not recorded, and hence all of the phenomena reported must have occurred at velocities below the threshold velocity. This cardinal point of impact behavior, velocity sensitivity, has not been covered by the work reported to date.

CLOSURE

It has seemed proper to consider this subject matter broadly so that emphasis could be placed on the principles rather than on the details of metal behavior. This applies particularly to the stress analysis of the notch effect.

PHYSICAL PROPERTIES OF FOUR LOW-ALLOY HIGH-STRENGTH STEELS

By W. L. Collins[1] and T. J. Dolan[1]

Synopsis

Tests were made to obtain information on the load-resisting properties of four low-alloy high-strength steels and one ordinary structural carbon steel when subjected to static, impact, and repeated loads. Results are presented from static load tests in tension, torsion, and notched tension tests and from tension tests following a prestressing. Charpy impact values obtained from bend tests of a V-notch specimen, and tension impact values for both notched and unnotched specimens tested in the Charpy machine are included. The endurance limits obtained from repeated bending fatigue tests on specimens in air and in a stream of water, for both solid specimens and specimens containing a hole or fillet, are given.

The results indicate that two of the low-alloy high-strength steels had considerably higher ratios of yield point to tensile strength than ordinary structural carbon steel and had about the same ductility as ordinary structural carbon steel. All steels tested had well-defined yield points as determined by drop of beam when gage lengths of 8 in. were used. The properties obtained from the impact tests apparently depend upon the type of specimen used. For repeated load specimens free from abrupt changes in section and tested in air, all four low-alloy high-strength steels had endurance limits at least 67 per cent greater than that of structural carbon steel. However, the low-alloy high-strength steels exhibited a greater fatigue notch-sensitivity, and were damaged to a greater extent by corrosion-fatigue than ordinary structural carbon steel. The values of the corrosion-fatigue endurance limits for specimens of all four low-alloy high-strength steels containing a hole or fillet were about the same and were small as compared with their endurance limits in air.

The great interest of engineers, metallurgists, and steel producers in the rather recently developed group of steels known as "low-alloy high-strength" steels is evident from the three symposiums[2,3,4] held by national engineering societies in 1936. The problems involved in the production, fabrication, and use of the new steels have been presented in the various symposiums. However, there is need for more information concerning the properties of these low-alloy high-strength steels as is well brought out in the following quotation from the Progress Report of Subcommittee No. 2, Committee on Steel, of the Structural Division of the American Society of Civil Engineers.[5] "Again and again, committee deliberation has brought forward questions and desires for exact knowledge that seem to have no answer because of lack of sufficient knowledge of the mechanical properties of these steels." The use of these steels in moving parts of various types of machines also calls for a knowledge of their notch-sensitivity in fatigue and under impact loading, their corrosion-fatigue properties, their sensitivity to strain hardening, and their ability to undergo large plastic yielding at points of stress concentrations as well as in a member as a whole.

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[1] Associate, and Assistant Professor, respectively, Department of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[2] Symposium on High-Strength Constructional Metals, Pittsburgh Regional Meeting, Am. Soc. Testing Mats. (1936). (Available as separate publication.)

[3] Symposium on Structural Applications of Steel and Light-Weight Alloys, *Proceedings*, Am. Soc. Civil Engrs., Vol. 62, No. 8, Part 1, October, 1936, p. 1125.

[4] Symposium on Role of Metals in New Transportation, *Metals Technology*, Vol. 3, No. 7, October, 1936, p. 3.

[5] *Proceedings*, Am. Soc. Civil Engrs., Vol. 62, No. 3, March, 1936, p. 384.

This paper presents information, obtained from laboratory tests of machined specimens, on the mechanical properties of four of the fifteen or twenty[6,7] new low-alloy high-strength steels developed in this country during the past few years, and of one ordinary structural carbon steel.

TABLE I.—ANALYSIS OF HEATS AS FURNISHED BY STEEL COMPANIES.

Material	Chemical Composition, per cent								Total Except Carbon, per cent
	Carbon	Manganese	Phosphorus	Sulfur	Silicon	Copper	Chromium	Nickel	
A........	0.08	0.27	0.145	0.020	0.80	0.41	1.01	....	2.655
B........	0.08	0.43	0.104	0.022	0.162	1.07	....	0.54	2.328
C........	0.22	0.69	0.045	0.019	0.25	0.92	....	1.98	3.904
D........	0.37	1.14	0.021	0.033	0.84	....	0.50	....	2.534
36........	0.21	0.43	0.008	0.028	.....	....	....	....	0.466

All the steels investigated were subjected to tension, compression, torsion, hardness, impact, and repeated-load tests. Tension specimens with and without notches were tested under both static and dynamic (impact) loads. The repeated load (fatigue) tests were made on specimens in air and also under a stream of tap water, the specimens being both with and without stress raisers (holes or fillets). The properties determined from these tests are considered to be of value primarily in connection with the use of the steels in load-resisting members, and are of only secondary importance in a study of fabricating and processing operations. The data for all the tests made are not included in this paper because of the limitations placed on its length.

Acknowledgment is made to F. B. Seely, H. F. Moore, and J. O. Draffin for their help and suggestions in planning the tests and in preparing this paper. The help of C. H. Dunn, S. D. Gralak, C. B. Mansky, J. A. Nachowitz, R. Richmond, L. E. Salch, J. A. Snyder, and R. B. Stampfle, students in the College of Engineering who carried out various parts of the investigation, mainly as theses during the past two years, in close cooperation with the authors, is also acknowledged. They are to be commended for their interest and the care with which their work was done.

MATERIALS AND SPECIMENS

Materials Tested.—The low-alloy high-strength steels tested have been designated as steels A, B, C, and D. The ordinary structural carbon steel is referred to as steel 36.[8] All the material was in the form of hot-rolled round bars having diameters of $\frac{3}{4}$ in. and all tests were made with the material in the as-rolled condition. The bars of each steel were rolled from one billet; the chemical compositions of the heats from which the billets came, as furnished by the steel mills, are given in Table I. The steels were selected to give a variety of alloying elements. Steels A and B were very low in carbon content and

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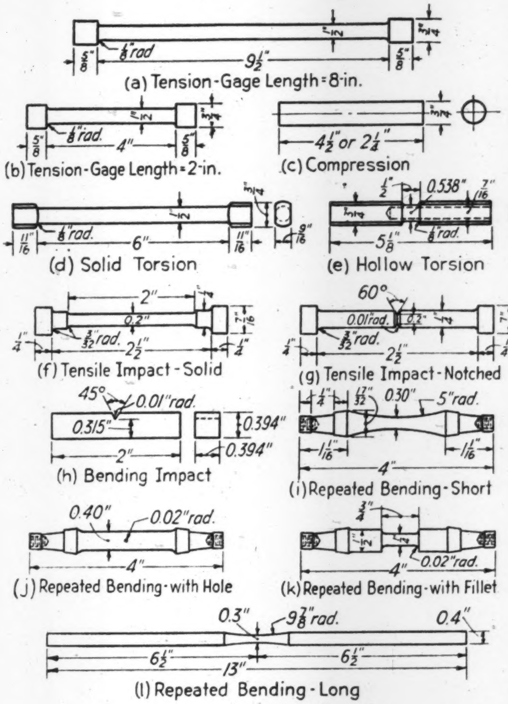
[6] E. F. Cone, "Carbon and Low-Alloy Steels," Symposium on High-Strength Constructional Metals, Pittsburgh Regional Meeting, Am. Soc. Testing Mats., p. 1 (1936). (Symposium available as separate publication.)

[7] H. W. Gillett, "Trends in the Metallurgy of Low-Alloy, High-Strength Structural Steels," *Metals Technology,* Vol. 3, No. 7 October, 1936, p. 40

[8] Standard Specifications for Steel for Buildings (A 9-36), 1936 Book of A.S.T.M. Standards, Part I, p. 9.



(a) Tension-Gage Length = 8-in.
9½"
⅝"
⅝" rad
(b) Tension-Gage Length = 2-in.
4"
⅝" rad.
(c) Compression
4½" or 2¼"
(d) Solid Torsion
6"
⅝" rad.
11/16"
9/16"
(e) Hollow Torsion
5⅝"
0.538"
7/16"
⅝" rad.
⅝"
(f) Tensile Impact - Solid
2"
0.2"
3/32" rad.
2½"
¼"
7/16"
(g) Tensile Impact - Notched
60°
0.01" rad.
0.2"
3/32" rad.
2½"
(h) Bending Impact
45°
0.01" rad.
0.315"
0.394"
0.394"
2"
(i) Repeated Bending - Short
17/32"
0.30"
5" rad.
1 1/16"
4"
(j) Repeated Bending - with Hole
0.40"
0.02" rad.
4"
(k) Repeated Bending - with Fillet
¾"
0.02" rad.
4"
(l) Repeated Bending - Long
0.3"
9⅞" rad.
0.4"
6½"
6½"
13"

Fig. 1.—Details of Test Specimens.

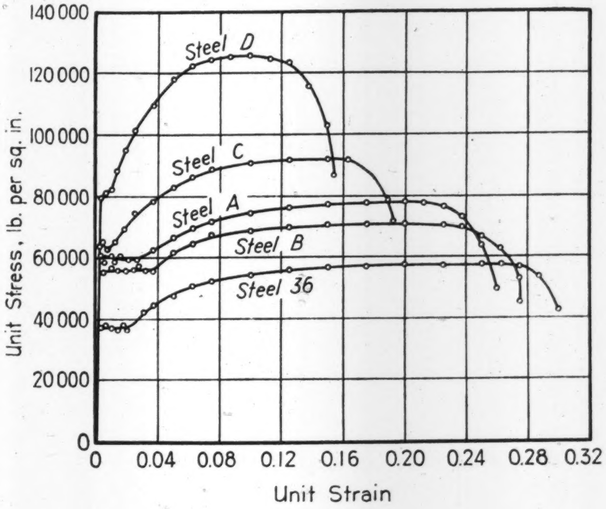


Fig. 2.—Typical Stress-Strain Diagrams of Tension Tests.

8-in. gage length. 0.5-in. diameter.

contained phosphorus and copper as alloying elements. Relatively large quantities of copper and nickel were found in steel C. Steel D had the highest carbon content and also contained considerable quantities of manganese and silicon as well as some chromium.

Description of Specimens.—The types of specimens used are shown in Fig. 1. Three tension specimens similar to (*a*) and (*b*) of Fig. 1 for steels A, B, and D had threaded ends instead of the shoulders shown. The compression specimens (*c*) were machined only on the ends. The tension impact specimens, (*f*) and (*g*), were polished with No. 00 emery paper; to insure breaking in the gage length the unnotched specimens had their diameters near the center about 0.001 in. less than at the ends. Repeated load specimens (*i*), (*j*) and (*l*) were polished longitudinally with No. 00 emery paper; the fillets on specimens (*k*) were left as turned and the straight portion of smaller diameter was finished with a file.

TABLE II.—STATIC TENSION TESTS.

8-in. gage length. Nominal diameter, 0.500 in.

Each value is the average of three tests, except as noted.

Material	Elastic Limit, lb. per sq. in.[a]	Yield Point, lb. per sq. in.	Tensile Strength, lb. per sq. in.	Elongation, per cent		Reduction of Area, per cent	Toughness, in-lb. per cu. in.[b]	Modulus of Elasticity, lb. per sq. in.	Ratio, Elastic Limit to Yield Point[d]	Ratio, Yield Point to Tensile Strength	Brinell Hardness
				8 in.	2 in.						
A.........	60 000	60 600	78 400	26.6	40.2	72.0	19 100	30 000 000	0.99	0.77	156
B.........	56 100	56 000	70 800	27.3	38.0	69.2	17 500	29 700 000	0.99	0.79	145
C.........	54 100	63 000	91 300	18.5	29.6	54.5	17 500	29 100 000	0.84	0.69	194
D.........	80 600	81 300	125 000	14.8	23.1	58.1	16 600	30 000 000	0.97	0.65	255
36[c]........	38 300	36 500	57 400	29.9	41.6	64.0	15 900	29 600 000	0.98	0.64	109

[a] For method of determination see page 164. Values are averages of two tests.

[b] Total energy required to rupture specimen as determined by the area under the entire stress-strain curve.

[c] Values are averages of four tests, except for elastic limit.

[d] Computed from the yield points of the specimens used to determine the elastic limit.

Three types of notched specimens were used in the static tests and were similar to the short tension specimens (*b*) in all respects except for a notch machined midway between 2-in. gage marks. The types of notch used were: (1) a V-notch 0.05 in. deep having a radius of 0.01 in. at the root, (2) a semicircular notch having a radius of 0.05 in. and a depth of 0.05 in., and (3) a sharp rectangular notch 0.05 in. deep and 0.100 in. long.

TYPES OF TEST AND TEST DATA

Static Tension Tests.—Six specimens of each material, three each of types (*a*) and (*b*), were tested to determine the static properties in tension. A typical stress-strain curve for each material is shown in Fig. 2 and the average values of properties as found from the 8-in. gage length specimens are given in Table II. The same properties were also determined for specimens having gage lengths of 2 in. but, except for elongation, are not included in this paper. The tests were carried out in accordance with the A.S.T.M. Standard Methods E 8 - 36.[9] The two specimens of each material used in the tests for elastic limits were of type (*a*), and were tested in addition to the other specimens. The stress-set curves obtained for determining elastic limits are not included in this paper.

Static Compression Tests.—Three long specimens $\left(\frac{l}{d} = 6\right)$ were used to determine moduli of elasticity in compression; three short ones $\left(\frac{l}{d} = 3\right)$ were used to determine yield points. No stress-strain curves or tabulated data for compression tests are given because the results differed so little from the corresponding tensile properties. The tests were made in a loading rig similar to that shown in Fig. 3 of the A.S.T.M. Tentative Methods E 9 – 33 T.[10]

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[9] Standard Methods of Tension Testing of Metallic Materials (E 8 - 36), 1936 Book of A.S.T.M. Standards, Part I, p. 833.

Static Torsion Tests.—Both solid and hollow specimens, as shown in Fig. 1 (*d*) and (*e*), were used for the static torsion tests. The average values of properties determined from three tests of each type of specimen are given in Table III. Stress-strain diagrams for shear (torsion) are not presented because they were all typical of those found for ductile steels and bore the same general relation to each other as did the tensile stress-strain curves shown in Fig. 2.

TABLE III.—STATIC TORSION TESTS.

Solid specimens: Nominal diameter, 0.500 in.; gage length, 5 in.
Hollow specimens: Nominal outside diameter, 0.538 in.; nominal wall thickness, 0.050 in.
Each value is the average of three tests.

Material	Yield point, lb. per sq. in.			Modulus of Rupture, lb. per sq. in.[a]		Modulus of Elasticity, Solid, lb. per sq. in.	Ratio, Yield Point (Hollow Specimen) to Modulus of Rupture (Hollow Specimen)	Ratio, Yield Point (Hollow Specimen) to Yield Point in Tension
	Solid	Hollow	Ratio, Hollow to Solid	Solid	Hollow			
A	39 800	31 900	0.801	85 500	59 000	11 900 000	0.540	0.527
B	38 800	36 700	0.946	77 300	62 900	11 500 000	0.584	0.655
C	46 400[b]	39 400	0.850	92 000	77 200	11 800 000	0.510	0.625
D	58 600[b]	51 000	0.870	113 000	95 200	12 200 000	0.536	0.627
36	25 400	21 700	0.897	65 100	55 000	11 900 000	0.398	0.595

[a] Value of s in torsion formula, $s = \frac{Tc}{J}$, when the maximum twisting moment is substituted for T.

[b] Yield Strength; offset 0.2 per cent. Yield point not well defined.

Bending and Tension Impact Tests.—The bending impact test made was the standard Charpy test using the V-notch specimen shown in Fig. 1 (*h*). The unnotched and notched specimens shown in Fig. 1 (*f*) and (*g*) were used for the tension impact tests, which were also made in the Charpy machine. It should be noted that the diameter at the root of the notches in the notched specimens was approximately equal to the diameter of the unnotched specimens. A special rig with spherical seats devised by S. W. Lyon, of the University of Illinois, was used to attach the tension specimens to the pendulum. The limitations of this holding apparatus made it impossible to test 2-in. gage lengths of constant diameter. However, since a 2-in. length encroached very little on the fillets it was felt that stating elongation in 2 in. was justifiable. The average impact values of three tests of each type of specimen for all five steels are presented in Table IV.

Repeated Loading.—The repeated load tests were made to determine: (1) the endurance limits of all five steels when subjected to completely reversed cycles of flexural stress in air, (2) the notch-sensitivity in fatigue as indicated by the reduction in flexural fatigue strength produced by a small hole or fillet in specimens of these steels, and (3) the corrosion-fatigue sensitivity as measured by the additional reduction in fatigue strength caused by applying a small stream of fresh water to specimens (of the same types as those tested in air) while they were being stressed.

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[10] Tentative Methods of Compression Testing of Metallic Materials (E 9 – 33 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 959 (1933); also 1937 Book of A.S.T.M. Tentative Standards, p. 1499.

The types of specimens used are shown in Fig. 1 (*i*), (*j*), (*k*), and (*l*). All tests were made in modified Farmer type rotating-beam machines producing 1450 completely reversed cycles of stress per minute. The nominal stress in all specimens was calculated by using the ordinary flexure formula; for the specimens with transverse holes no allowance was made for the material removed to form the hole. The surfaces of the specimens used in the corrosion-fatigue tests were washed with gasoline and acetone to remove all traces of oil before testing. The water used was fresh tap water having a pH value of 7.6 and was piped directly to the machines, a small stream flowing on each specimen while it was being stressed.

TABLE IV.—TENSION AND BENDING IMPACT TESTS, CHARPY MACHINE.

Each value is the average of three tests, except as noted.

Material	Tension Impact						Charpy Notched Bending Test, ft-lb.[c]
	Energy to Rupture, ft-lb.		Ratio, Energy to Rupture for Notched Specimen to Energy to Rupture for Solid Specimen	Elongation in 2 in., per cent		Ratio, Elongation for Notched Specimen to Elongation for Solid Specimen	
	Solid[a]	Notched		Solid[a]	Notched		
A.......	129.1	42.0[b]	0.325	28.2	7.7	0.273	[d]
B.......	115.6	30.7	0.265	27.5	5.2	0.189	112
C.......	109.7	49.8	0.454	19.2	6.2	0.323	24.4[b]
D.......	113.6	56.4	0.496	15.5	6.7	0.432	13.4[b]
36.......	107.5	25.3	0.235	31.5	4.5[b]	0.143	[d]

[a] Solid specimens are those without abrupt change in section, as shown in Fig. 1 (*f*).

[b] Average of two tests.

[c] V-notch specimen, as shown in Fig. 1 (*h*).

[d] Specimens did not break, but were bent and carried through the machine by the pendulum.

A typical set of *S-N* curves for steel D is shown in Fig. 3, and a summary of the endurance limits scaled from curves of this type is given in Table V. The corrosion-fatigue tests were run for a minimum of 40 million cycles, and in some cases to 100 million cycles, to locate fairly definite horizontal asymptotes to the *S-N* curves.

Static Notch-Sensitivity Tests.—All notches in the specimens used for determining notch-sensitivity, except those for steel 36, were machined at the same time and with the same tools. For each material six specimens were tested, two with V-notches, two with semicircular notches, and two with rectangular notches. The average values of yield point, tensile strength, and elongation in 2 in. for the V-notch specimens are given in Table VI; the data for the other types of notches are not presented.

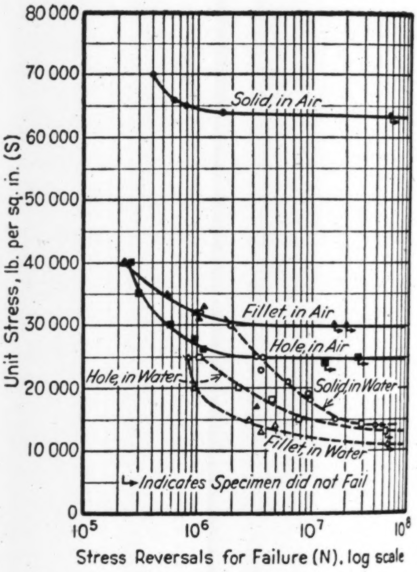

FIG. 3.—*S-N* Diagrams for Hot-Rolled Steel D for Complete Reversals of Bending Stress.

Static Overstrain Tests.—The effects of overstrain on the physical properties of the steels were studied by prestressing specimens of the type shown in Fig. 1 (*a*) and then immediately testing them to failure. In general, three specimens of each steel were tested, one specimen being subjected to each of the following prestresses: (1) the yield point, (2) midway between the yield point and tensile strength, and (3) at about 95 per cent of the tensile strength. For most of these tests the diameter of the specimen was measured for each increase of load beyond the yield point and readings of load and deformation were taken at frequent intervals during the application and release of all loads, but these results are not discussed in this paper.

TABLE V.—REVERSED FLEXURAL FATIGUE PROPERTIES.

NOTE:

$$K = \frac{\text{Endurance limit of solid specimen tested in air}}{\text{Endurance limit of specimen with stress raiser tested in air}}$$

$$K_c = \frac{\text{Endurance limit of specimen tested in air}}{\text{Endurance limit of same type of specimen tested in water}}$$

Total stress concentration factor = $K \times K_c$.

Material	Type of Specimen[a]	Endurance Limit, lb. per sq. in.		Stress Concentration Factor (See note)		Ratio, Endurance Limit to Tensile Strength
		In Air	In Water	K	K_c	
A	Solid	54 000	29 000	1.00	1.86	0.689
	Fillet	24 500	13 000	2.20	1.88	
	Hole	25 000	18 000	2.16	1.39	
B	Solid	50 000	24 000	1.00	2.08	0.707
	Fillet	24 000	14 000	2.08	1.72	
	Hole	23 800	17 000	2.10	1.40	
C	Solid	54 500	19 000	1.00	2.87	0.593
	Fillet	26 000	15 000	2.10	1.73	
	Hole	26 500	16 000	2.06	1.66	
D	Solid	63 500	14 000	1.00	4.53	0.508
	Fillet	30 000	11 000	2.12	2.73	
	Hole	25 000	13 000	2.54	1.92	
36	Solid	29 500	19 000	1.00	1.55	0.513

[a] Solid specimens are those without abrupt change in section, as shown in Fig. 1 (*i*) and (*l*).

Hardness Tests.—Brinell, Rockwell, and Shore scleroscope hardness tests were made on the rough polished flat ends of specimens. The average results of three or more Brinell tests (3000-kg. weight) of each material are given in Table II.

Metallographic Structure.—Both longitudinal and transverse sections of the $\frac{3}{4}$-in. round bars were examined under the microscope after having been polished and etched in a 2 per cent Nital solution. At a magnification of 200 diameters, the photomicrographs of the longitudinal sections of steels A, B, and 36 showed a banded structure, but, as was verified by the photomicrographs of transverse sections, there was an even distribution of constituents throughout the cross-section. All five steels appeared to be of good quality and free from large non-metallic inclusions. The structures were normal for the as-rolled condition, due consideration being given to the chemical compositions.

TABLE VI.—STATIC TENSION TESTS OF V-NOTCH SPECIMENS.

Gage length = 2 in. Nominal diameter of specimen = 0.500 in.

Nominal diameter at root of notch = 0.400 in.

Each value is the average of two tests.

Material	Yield Point		Tensile Strength		Elongation in 2 in.	
	At Root of Notch, lb. per sq. in.	Ratio, Yield Point at Root of Notch to Yield Point of Solid[a] Specimens Having 2-in. Gage Lengths	At Root of Notch, lb. per sq. in.	Ratio, Tensile Strength at Root of Notch to Tensile Strength of Solid[a] Specimens Having 2-in. Gage Lengths	Per Cent	Ratio, Elongation of Notched Specimens to Elongation in 2 in. for Solid[a] Specimens
A	84 000	1.45	106 000	1.38	12.5	0.312
B	79 600	1.43	97 100	1.36	11.0	0.289
C	99 300	1.59	132 000	1.44	6.2	0.209
D	117 000	1.42	160 000	1.25	4.5	0.196
36	51 800	1.46	82 100	1.38	10.0	0.241

[a] "Solid" specimens are those without abrupt change n section, as shown in Fig. 1 (*b*).

Examination of the steels at 900 diameters disclosed uniform grains of ferrite and a well-defined laminar structure of the pearlite for steels A, B, and 36. Steels C and D contained, in addition to ferrite and pearlite, a small amount of sorbite.

ANALYSIS OF TEST DATA

In studying the data presented in the tables, emphasis will be placed on the comparison of the properties of the low-alloy high-strength steels with the properties of steel 36. The data in Table II indicate that steel 36 did not satisfy the tensile strength requirement (60,000 to 72,000 lb. per sq. in.) of the specification under which it was purchased, but did satisfy the 1933 specification for similar material. It is felt that the failure to meet the tensile strength requirement does not detract seriously from the validity of any comparisons made.

Static Tension Test Data.—A study of the data of Table II and of the stress-strain curves shown in Fig. 2 reveals that all five steels had well-marked yield points as determined by drop of beam when gage lengths of 8 in. were used. The amounts of elongation at the yield point varied greatly for the different steels, being least for steel D and greatest for steel B (see Fig. 2). The magnitudes of these elongations may be of significance in determining the ability of the steels to undergo deep drawing operations.[11]

From Table II it can be seen that steels A and B had high ratios of yield point to tensile strength, 0.77 and 0.79, respectively. Such high ratios suggest the possibility of using high working stresses under static loads, but a discussion of the significance of such high ratios for any given use is outside the scope of this paper.

It should also be noted that the elongation after rupture (Table II) in both 8-in. and 2-in. gage lengths for steels A and B were comparable to the values for steel 36. Since these same two steels had greater reductions of area than steel 36, it can be said that, as determined by these two conventional measures of ductility, steels A and B are about as ductile as steel 36. By the same criteria, steels C and D are distinctly less ductile than steel 36.

For steels A, B, D, and 36 there were no measurable sets until their yield points were reached; in steel C measurable set occurred at about 84 per cent of the yield point. In determining the elastic limits it was realized that there was much uncertainty in selecting points of tangency on the stress-set curves, the points of tangency representing the maximum stress corresponding to no set, which, by definition, is the elastic limit. The elastic limit therefore was selected as the unit-stress corresponding to a permanent set of 0.00005 in. per inch after removal of the load, this value of set being slightly greater than that corresponding to one-half the smallest direct reading of the extensometer and thus being a reproducible value.

The average yield points, tensile strengths, and moduli of elasticity determined from specimens having gage lengths of 2 in. were within 5 per cent of the values found from specimens having 8-in. gage lengths. The yield points generally were a little lower and the ultimate strengths a little higher for the specimens having 2-in. gage lengths.

Static Compression Test Data.—The yield points and moduli of elasticity determined in compression were from 4 to 8 per cent higher than the corresponding values in tension, the gage lengths being 2 in. in compression and 8 in. in tension. The results are thus in general agreement with other tests[12] made to determine the relationship between tensile and compressive properties of steel. The yield points found for unmachined specimens having a ratio of length to diameter equal to 6 were very nearly the same as for specimens in which the ratio of length to diameter was 3.

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[11] J. Winlock and R. W. E. Leiter, "Some Factors Affecting the Plastic Deformation of Sheet and Strip Steel and Their Relation to the Deep Drawing Properties," *Transactions*, Am. Soc. Metals., Vol. XXV, No. 1, March, 1937, p. 163.

[12] F. B. Seely and W. J. Putnam, "The Relation Between the Elastic Strengths of Steel in Tension, Compression and Shear," *Bulletin No. 115*, Engineering Experiment Station, University of Illinois (1919).

Static Torsion Test Data.—The low ratio of the torsional shearing yield point of the hollow specimen to that of the solid specimen for steel A could not be explained (see Table III). For the other steels tested, the value of this ratio varies but little and is in accord with the results of other investigations.[12] Table III indicates that for the hollow specimens the yield points of the low-alloy high-strength steels were considerably larger in proportion to their moduli of rupture than was found for steel 36. The values of the ratios were much less than for the ratios of tensile yield points to tensile strengths given in Table III; both ratios, however, were greatest for steel B and least for steel 36. The ratios of shearing yield point to tensile yield point, Table III, were about the same (0.595 to 0.655) for all the steels tested except steel A which had a ratio of 0.527.

Impact Resistance.—The data in Table IV show that the Charpy tension impact values for solid (unnotched) specimens of all steels tested were relatively about the same, as were also the values of total energy (designated as toughness in Table II) required to rupture the tension specimens in the static load tests. The bend test, using the V-notch specimen, made the greatest distinction between materials, with the notched tension impact test next in order. However, the arrangement of the five steels in the order of impact resistance, as determined in a Charpy machine, depended almost entirely upon the type of specimen used.

The values of elongation in 2 in. for unnotched specimens $\left(\frac{l}{d} = 10\right)$ subjected to tension impact loads were slightly greater than the elongations determined from specimens subjected to static loads when 8-in. gage lengths $\left(\frac{l}{d} = 16\right)$ were used. Since the elongation is dependent upon the ratio $\frac{l}{d}$ (gage length divided by diameter of specimen) it is believed that for the same ratio of $\frac{l}{d}$ the elongations under impact and static loads would be approximately equal.

The maximum speed of the pendulum of the Charpy machine was about 17 ft. per sec. No tests were made at very high speeds of testing, but, as has been shown by Mann,[13] very high velocities of loading may have a marked effect on the test values obtained under impact loading.

Repeated Bending Test Data.—In comparing the endurance limits obtained for the solid specimens (see Table V for definition) tested in air, it will be noted that the endurance ratios[14] for steels A and B are 0.69 and 0.71, respectively. These ratios are much higher than the value of about 0.5 which was found for steels D and 36 and which is usually considered to apply to the more widely used carbon steels. When tested in air, the endurance limits of the solid specimens of the low-alloy high-strength steels (Table V) were all at least 67 per cent greater than that of steel 36. In general, the endurance limits of the low-alloy high-strength steel specimens containing a hole or fillet, and tested in air, were fairly close in numerical value, ranging from about 24,000 to 30,000 lb. per sq. in. Similarly, the stress concentration factors, K, (Table V) for a hole or a fillet in specimens of the four low-alloy high-strength steels tested in air did not vary a great deal.

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[13] H. C. Mann, "High-Velocity Tension-Impact Tests," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 85 (1936).

[14] Endurance ratio is the ratio of the endurance limit of the solid specimen tested in air to the static tensile strength of the material. Values are listed in Table V.

The values of K in Table V are considerably higher than the results obtained from fatigue tests of steels by other investigators as reported by Peterson and Wahl.[15] For fillets (of about the proportions used in the present tests) in ordinary carbon steels they show values of K ranging from 1.45 to 1.85, whereas for various alloy steels their values of K range from about 1.68 to 2.17. It seems likely, therefore, that the low-alloy high-strength steels tested exhibit a greater fatigue notch-sensitivity than most ordinary low-carbon steels. For steel D the stress concentration factor of 2.54 for the specimen with a hole is considerably larger than that obtained from the tests of any of the other low-alloy high-strength steels. Steel D apparently has a greater fatigue notch-sensitivity to this type of discontinuity than the other steels tested, but the stress concentration factor is no higher than has been obtained for some heat-treated alloy steels.[16]

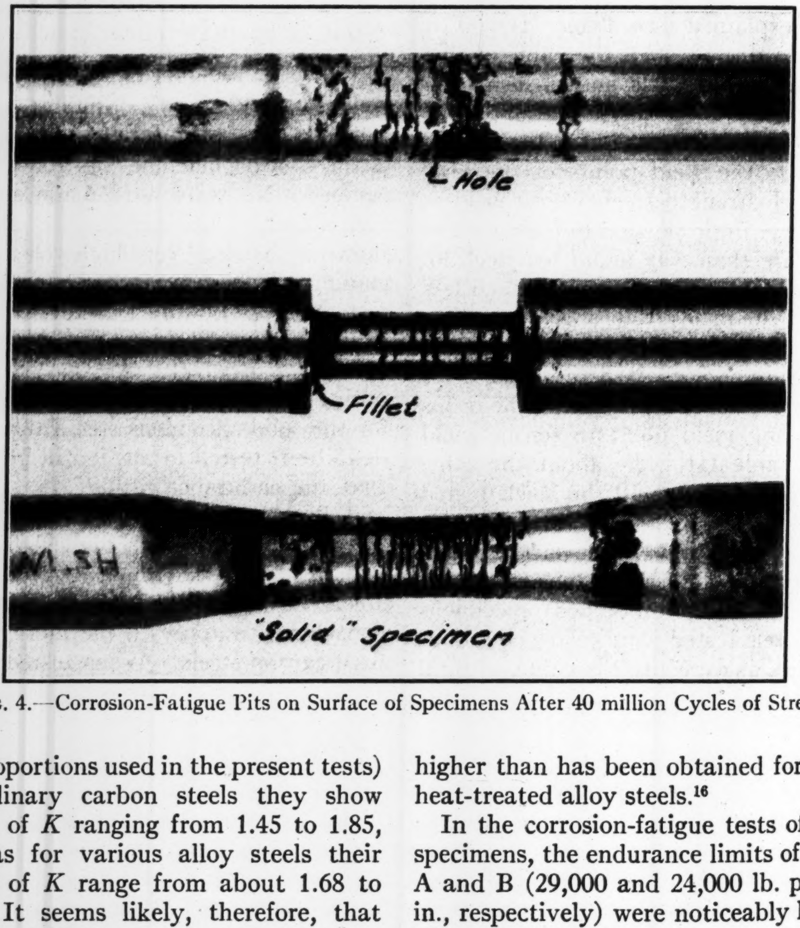

FIG. 4.—Corrosion-Fatigue Pits on Surface of Specimens After 40 million Cycles of Stress.

In the corrosion-fatigue tests of solid specimens, the endurance limits of steels A and B (29,000 and 24,000 lb. per sq. in., respectively) were noticeably higher than those of the other steels tested. It is of interest to note that these two steels were the ones containing more

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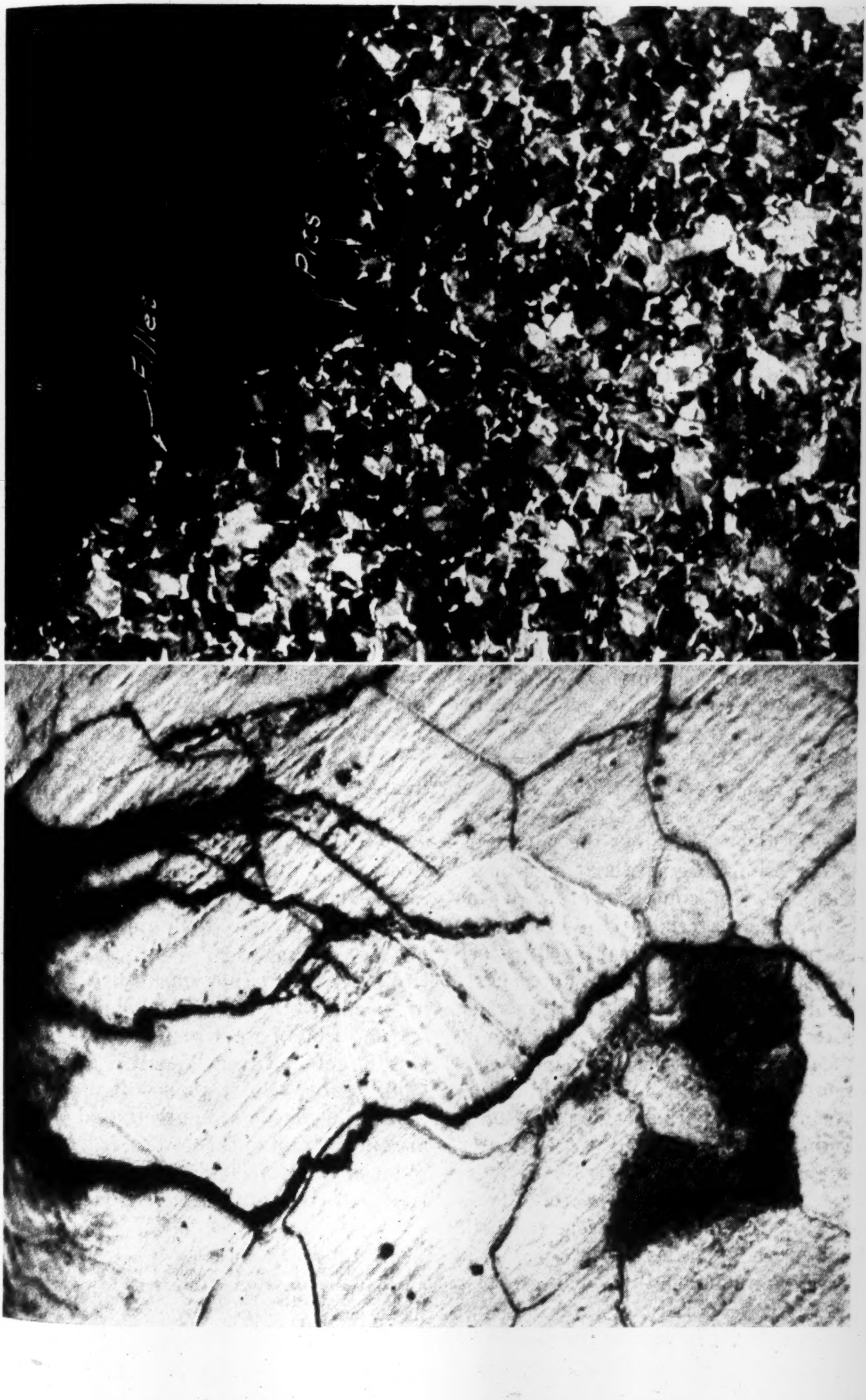
[15] R. E. Peterson and A. M. Wahl, "Two- and Three-Dimensional Cases of Stress Concentration, and Comparison with Fatigue Tests," *Journal of Applied Mechanics*, Vol. 3, No. 4, p. 415 (1936).

[16] T. J. Dolan, "Simultaneous Effects of Corrosion and Abrupt Changes in Section on the Fatigue Strength of Steel," *Journal of Applied Mechanics*, December 1938.



(a) Steel B (× 2200).

(b) Steel D (× 200).

FIG. 5.—Corrosion-Fatigue Pits on Longitudinal Sections of Specimens. Etched in 2 per cent Nital.

than 0.10 per cent phosphorus as an alloying element. However, for specimens containing a hole or fillet, and tested in water, there appeared to be little difference in the fatigue strengths of specimens of the four low-alloy high-strength steels.

Figure 4 shows the typical deep corrosion fatigue pits formed on the surface of some of the specimens of steels B and C, all the specimens having been subjected to more than 40 million cycles of bending stress without producing a fatigue crack that led to fracture of the specimen. It is apparent that these pits generally lie on the planes of maximum flexural stress and act as stress raisers forming incipient cracks that lead to complete failure. Most of the corrosion products were removed from the surface of these specimens by rubbing with No. 00 emery paper before taking the photograph. Probably these corrosion products embedded in the pits aided in inhibiting further corrosion, and thereby helped to prevent complete failure of the specimens at the lower stresses.

Figure 5 shows longitudinal planes along the center lines of specimens of steels B and D and shows the nature of these corrosion-fatigue pits at high magnifications. The complex nature of the cracks in the material at the bottom of these pits is clearly apparent for steel B; for steel D there were usually sharp pointed pits with only a single crack radiating from the bottom of the pit.

A comparison of the corrosion stress concentration factors, K_c, (Table V) indicates that the solid specimens of steels C and D were damaged to a greater extent than the other steels by the corrosive action of the tap water. It is interesting to note that steel D, which exhibited the greatest endurance limit in air, was damaged to the greatest extent by the corrosive action of tap water; for all three types of specimen indicated in Table V this steel actually had the lowest corrosion-fatigue limits of any of the steels tested.

A comparison with the results of other investigations, however, indicates that the fatigue strengths obtained for steel D are not abnormally low. Slightly lower corrosion-fatigue limits than those listed for steel D have been obtained[16] (using the same three types of specimens as in the present tests) in tests of a heat-treated chromium-nickel steel which had a very high endurance limit (90,000 lb. per sq. in.) for solid specimens in air. It is probable that steel 36 would exhibit about the same strength as the low-alloy high-strength steels if tested under the particularly damaging combination of corrosion-fatigue and localized stresses at abrupt changes in section. Some evidence to this effect was obtained from similar corrosion-fatigue tests in which the stress was reversed in torsion and the specimen contained a small hole or fillet.[17] The endurance limit of a low-carbon steel (S.A.E. No. 1020) was practically the same as that of heat-treated chromium-nickel (S.A.E. No. 3140) steel. Tests similar to those made on the low-alloy high-strength steels are contemplated for steel 36 and repeated load tests for tensile stresses varying from zero to a maximum are now in progress.

Notch-Sensitivity Under Static Loading.—The data in Table VI show that in static tests of specimens having gage lengths of 2 in., a V-notch increased considerably the values of the yield point and tensile strength (based on the area at the root of the notch), and greatly reduced the elongation. Whether sensitivity to the notch is based on increase in strength or decrease in elongation, the steels were not greatly different in sensitivity, although steel D showed somewhat greater sensitivity to the notch than the other four steels.

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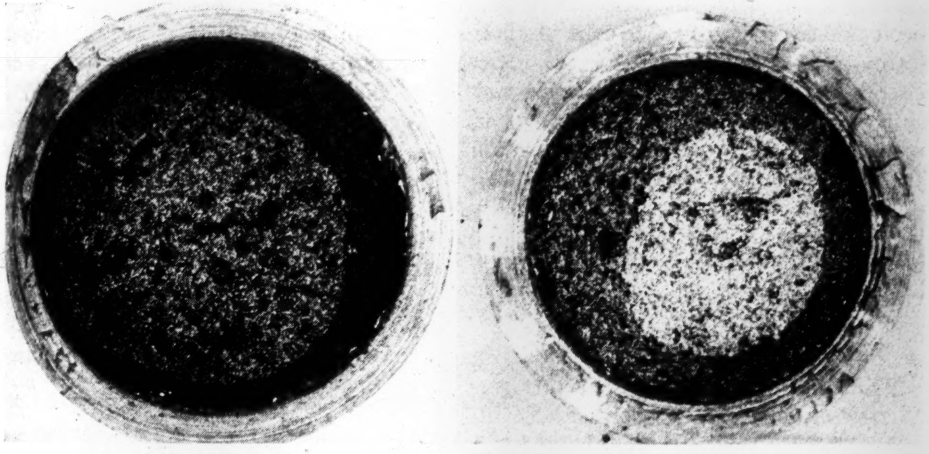
[17] T. J. Dolan, "Combined Effect of Corrosion and Stress Concentration at Holes and Fillets in Steel Specimens Subjected to Reversed Torsional Stresses," *Bulletin No. 293*, Engineering Experiment Station, University of Illinois (1937).

The V-notch increased the unit stress at the yield point and ultimate strength more than did either the semicircular or rectangular notches, the stresses being based on the original areas at the roots of the notches. The fact that the V-notch influenced the behavior of the specimen more than the semicircular notch (and the semicircular more than the rectangular) is probably due to the material at the root of the V-notch being restrained more from lateral movement than similarly located material in the other types of notched specimens; in fact, a measurable reduction of area was found for the specimens with rectangular and semicircular notches.

Another observable phenomenon in the static tests of the V-notch specimens was the formation and opening of cracks at the roots of the notches as the tests progressed beyond the yield points. These cracks were observed in all steels except steel D. The extent to which the crack had penetrated into the interior could be detected by examination of the ruptured surface after final fracture. The difference in surface appearance of the cracked area can be noted in Fig. 6, which shows the area of fracture of V-notch specimens of steels C and 36. As can be seen, the cracks did not reduce the areas of all the steels tested by equal amounts. Further study of this action is being made.



(*a*) Steel C. (*b*) Steel 36.

FIG. 6.—Unetched Areas of Fracture of V-Notch Specimens Subjected to Static Loads (× 7).

Notch Sensitivity Under Impact Loading.—Notch sensitivity under tension impact loads, as indicated in the fourth and seventh columns of Table IV, is greater for steel 36 than for the low-alloy high-strength steels. There is considerable variation in the values for the low-alloy high-strength steels, and one of them, steel B, gave values quite close to those for steel 36.

Effect of Overstrain.—The effects of overstrain, as determined from the results of specimens tested to failure immediately after prestressing, were about the same for all five steels. In the following discussion, all stresses are based on the original cross-sectional areas of the specimens. Prestressing the five steels to the yield point had no effect on the yield point, the tensile strength, or the elongation in 8 in., as determined for the second application of load. Prestressing to midway between the yield point and tensile strength increased the yield point (which was not as well marked as on the initial application of load) to a value slightly higher than the value of the prestress; there were no changes in tensile strength or elongation. Since the stress-strain curves were so flat at high stresses, it is doubtful if it can be said that a yield point existed after the specimens had been prestressed to 95 per cent of their ultimate strengths. However, the change in rate of increase of load was sufficiently great to cause a drop of beam and the stress at the load at which the drop took place was called the yield point. This value was usually slightly less than the value of the prestress; again, no effect on tensile strength or elongation was observed.

CONCLUSIONS

The following general conclusions from the tests reported seem justified:

1. All five steels had well-defined tensile yield points, but the elongations at the yield points varied greatly, steel B having the greatest elongation and steels C and D the least (see Fig. 2).

2. The ductility of steels A and B, based on elongation and reduction of area, was about the same as for steel 36, whereas steels C and D were distinctly less ductile than steel 36. The elongation in 8 in. ranged from 29.6 per cent for steel 36 to 14.8 per cent for steel D.

3. The ratios of tensile yield points to tensile strengths for steels A and B were considerably higher (0.77 and 0.79, respectively) than for steels C, D, and 36, the values for the latter three being 0.69, 0.65, and 0.64, respectively.

4. The tensile elastic limit (unit stress at a set of 0.00005 in. per inch) of each of the five steels was approximately equal to the yield point of the same steels, except for steel C which had an elastic limit of only 0.84 of its yield point (see Table II).

5. The tensile moduli of elasticity of all five steels were practically equal to 30,000,000 lb. per sq. in., the range being from 29,100,000 to 30,000,000 lb. per sq. in. The torsional (shearing) moduli of elasticity varied only slightly from 12,000,000 lb. per sq. in.

6. The torsional (shearing) yield points as found from hollow specimens were approximately 0.90 of those found from solid specimens, except for steel A in which case the value was 0.80. The range in values was from 0.80 to 0.95.

7. The average value of the ratios of the torsional (shearing) yield points, as found from hollow specimens, to the tensile yield points for all the steels except steel A was 0.63; the value for steel A was 0.53 and the range in values was from 0.53 to 0.65.

8. The elongation in a 2-in. gage length $\left(\frac{l}{d} = 10\right)$ in specimens free from notches as obtained from the Charpy tension impact test was, for each of the five steels, slightly greater than the elongation determined for 8-in. gage lengths $\left(\frac{l}{d} = 16\right)$ when tested under static loads. It is believed that for equal ratios of $\frac{l}{d}$ the elongations would have been approximately the same for both types of loading.

9. V-notches 0.025 in. deep caused large decreases in elongation in 2 in. and in the energy required for rupture in the Charpy tension impact tests, but the decreases varied considerably with the different steels; the greatest

decrease occurred in steel 36 and the least in steel D (see Table IV).

10. A V-notch 0.05 in. deep caused a large decrease in the elongation in 2 in. and a large increase in tensile strength in the static tension test, but all five steels were affected approximately to the same extent.

11. The fatigue endurance limits for completely reversed bending stress in specimens of steels A and B that were free from abrupt changes in section and tested in air were about 0.70 of the tensile strength of the steels, as compared with a similar value of 0.50 for the ordinary structural carbon steel. Steel D also had a value of approximately 0.50 and steel C had a value of 0.59.

12. The fatigue strengths of all the low-alloy high-strength steels tested were reduced more than 50 per cent by a small hole or a small fillet in the specimen. They have, therefore, a relatively high degree of fatigue notch-sensitivity.

13. Likewise, corrosion caused by tap water running on the specimen while it was being repeatedly stressed resulted in a large decrease in the fatigue strength of the low-alloy high-strength steels tested; the water produced a greater decrease in the endurance limits of solid specimens of steels C and D than that produced by a small hole or fillet in specimens tested in air. The values of the corrosion-fatigue endurance limits, for specimens containing a hole or a fillet varied from 11,000 to 18,000 lb. per sq. in., whereas the endurance limits of the same low-alloy high-strength steels obtained from specimens free from abrupt changes in section and free from corrosion ranged from 50,000 to 63,500 lb. per sq. in.

DISCUSSION

Mr. R. F. Passano.[1]—It has occurred to me that the endurance limit might be associated with the yield point rather than with the tensile strength in steels showing elongation at the yield point.

In the following table, I have arranged the five steels of the paper in order of their elastic ratios:[2]

Material	Yield Point, lb. per sq. in.	Tensile Strength, lb. per sq. in.	Endurance Limit in Air, lb. per sq. in.	Ratio, Yield Point to Tensile Strength	Ratio, Endurance Limit to Tensile Strength	Ratio, Endurance Limit to Yield Point
B.......	56 000	70 800	50 000	0.791	0.707	0.893
A.......	60 600	78 400	54 000	0.774	0.689	0.891
C.......	63 000	91 300	54 500	0.690	0.593	0.865
D.......	81 300	125 000	63 500	0.650	0.508	0.781
36......	36 500	57 400	29 500	0.636	0.513	0.808
			Average	....	0.602	0.848

I call your attention to the way in which the endurance ratio parallels the elastic ratio (columns 5 and 6 of table). The one steel out of line is steel D: it has a definite yield point, but is practically without elongation at the yield point (see statement of proposition above). Its endurance limit is in accord with the old rule that the endurance limit is about 50 per cent of the tensile strength. Since it does not satisfy the qualifications of the proposition, let us consider further the four steels which are within the qualifications of the proposition.

The average endurance ratio for steels B, A, C, and 36 is 0.626, and the range of the four ratios is 0.194. The average endurance-yield point ratio is 0.864 with a range of 0.085 for the four observations. A ratio of 0.864 with a range of 9.8 per cent is a better relation than one of 0.626 with a range of 31.0 per cent.

I scarcely believe that the proposition has become a theorem by this simple argument.

When one makes fatigue tests, regularity of surface and section is required to obtain uniform fiber stress. When one makes corrosion tests, an irregularity of surface is required to obtain uniformity of attack. The smooth, polished surface used on fatigue specimens is a poor surface on which to make corrosion tests. On a polished surface, attack begins at spots: almost always such attack results in pits of a sort. The unattacked bright area of the specimen is available as cathode, and the fewer the spots of attack, the greater the penetration will be at the small anodic spots.

It would be interesting and perhaps worth while to prepare the surface of corrosion-fatigue specimens as though they were corrosion specimens (they are now prepared as though they were fatigue specimens). A surface capable of giving reproducible weight losses in an underwater corrosion test might behave under corrosion-fatigue quite differently from a surface that corrodes only at an indefinite and irregular number of spots.

Mr. D. J. McAdam, Jr.[3]—I wish to emphasize that the "endurance limits" reported for corrosion-fatigue conditions apply only to the times and cycle frequencies under which they were obtained. The so-called corrosion-fatigue limits are not absolute limits. The slower the cycle frequency and the longer the corrosion time, the lower is the stress required to cause the same amount of damage. At high cycle frequency, the curve of corrosion-fatigue may appear to be approaching a horizontal asymptote. If continued far enough, however, the curvature would reverse, and the curve would descend rapidly. The apparent approach to a horizontal asymptote probably is due to the balancing of two opposing factors: work hardening of the metal around the corrosion pits, and advance of the corrosion pit.

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[1] Research Engineer, The America Rolling Mill Co., Middletown, Ohio.

[2] Yield point divided by tensile strength.

[3] Metallurgist, National Bureau of Standards, Washington, D. C.

In regard to Mr. Passano's remarks, I would say that slight roughness of the surface prior to test probably is of much less importance in corrosion-fatigue than it is in ordinary corrosion. Corrosion-fatigue involves simultaneous action of stress and corrosion. The stress accelerates the corrosion and tends to change the form of the corrosion pits, making them sharper and deeper. The effect of these sharper and deeper pits on the fatigue limit greatly outweighs any effect of slight initial roughness of the surface.

MR. O. E. HARDER[4] (*by letter*).—A brief discussion of results which have been obtained at Battelle Memorial Institute on specimens of steel of the composition "B" in the authors' paper seems to be in order.

Corrosion-fatigue tests have been made on this steel in the "as-rolled" and the normalized condition. Tests have also been made on a steel of this same composition but of a higher carbon content (0.18 per cent) as normalized. The tests were run at about 40 r.p.m. and at a stress of about 30,000 lb. per sq. in. in a synthetic brine[5] with H_2S as was used by Bayless[6] in corrosion-fatigue tests. The brine was circulated by natural gas. These tests were run to determine the life of the specimen under the conditions stated. The load of 30,000 lb. per sq. in. was at one time considered about the maximum working stress for sucker rods. Bayless has reported similar tests on several steels and his data are included in the following table for comparison:

CORROSION-FATIGUE LIFE OF STEELS IN BRINE AND H_2S AT A STRESS OF ABOUT 30,000 LB. PER SQ. IN.

Material	Heat Treatment	Stress, lb. per sq. in.	Cycles to Cause Rupture
DATA BY BATTELLE			
B	As rolled	30 000	1 033 000
	As rolled	30 670	997 000
	Normalized	30 000	1 380 000
High carbon—B steel[a]	Normalized	30 000	1 207 400
0.08% C, max, 3½% Ni, 0.20% Mo	Normalized	30 000	1 365 000
DATA BY BAYLESS			
Low carbon, 3½% Ni-Mo steel	Normalized, quenched and drawn	30 000	1 266 130
3% Ni wrought iron	Normalized	30 000	850 050
0.31% C, 0.57% Ni, 1.27% Cu	Quenched and drawn	30 000	790 000
0.14% C, 2% Mn steel	Quenched and drawn	30 000	737 300
S.A.E. No. 1050	Normalized, quenched and drawn	30 000	702 400
Engine bolt refined plain wrought iron	None	30 000	330 700

[a] 0.189 per cent carbon, 0.65 per cent manganese, 0.186 per cent silicon, 0.026 per cent sulfur, 1.10 per cent copper, 0.67 per cent nickel.

The above data indicate that steel B has a longer life when tested in the normalized condition than as rolled and that about doubling the carbon content does not increase its life. It is of interest to note that the low-carbon materials such as steel B and 3½ per cent nickel-molybdenum steel had longer lives in this test than the S.A.E. No. 1050

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[4] Assistant Director, Battelle Memorial Inst., Columbus, Ohio.

[5]

NaCl	22 000 p.p.m.
$CaCl_2$	5 000 p.p.m.
$MgCl_2$	3 000 p.p.m.

[6] F. B. Bayless, "Sucker Rod Fatigue Changes with the Nature of Corrosive Elements in Oil," *Oil and Gas Journal*, August 15, 1935, pp 26–30.

steel which had been normalized, quenched and drawn. The agreement in the tests on the low carbon, $3\frac{1}{2}$ per cent nickel-molybdenum steel in the two laboratories is quite good.

As is suggested by the data in the paper and by the results reported above, the subject of corrosion-fatigue is complicated and much additional research is needed.

Acknowledgment is made to the Inland Steel Co. and Battelle Memorial Institute for permission to present the results in this discussion.

MESSRS. W. L. COLLINS[7] AND T. J. DOLAN[7] (*authors' closure by letter*).—The tabulation of the data as presented by Mr. Passano does show a fairly constant ratio of endurance limit to yield point for the steels listed. However, it is extremely doubtful whether this ratio would be anywhere near the same value for other steels, especially if the steels were given some heat treatment after being "hot-rolled." For instance, in comparing the properties of hot-rolled S.A.E. No. 3140 with those of the same material after quenching and tempering, the authors found that the endurance limit and tensile strength were both increased about 30 per cent by the heat treatment, whereas the yield point was raised about 95 per cent. For this heat-treated alloy steel a very definite elongation was observed at the yield point, but the ratio of endurance limit to yield point was only 0.576 as compared with the average of 0.848 for steels A, B, C, D and 36. The data reported by Moore and Kommers[8] on fifteen different steels indicated that no simple relation existed between the endurance limit and the "elastic limit," however determined. The fact that the endurance limits ranged from about 50 per cent to 150 per cent of the elastic strengths of these materials defied any attempts at such a correlation. However, it was found that the ultimate tensile strength or the Brinell hardness number seemed to furnish a better index of the endurance limit than any other property.

In general, it may be assumed that the endurance limit of a steel is a property that is more closely associated with fracture than it is with general yielding, but there is no reason to expect a close correlation of endurance limit with any other physical strength property of a material. The phenomena which govern the strength, or control the failure, of a material under repeated loads are somewhat different from those encountered under static loads.

With regard to the formation of corrosion pits, the authors have found that in several tests there were a large number of small pits on the highly stressed specimens, and a smaller number of longer pits on the specimens tested at lower stresses. This, however, may be due to the fact that the highly stressed specimens were run for a relatively short period of time and the corrosion pits consequently did not have sufficient time for much growth. Failures of members in service indicate the same types of corrosion pits as those found on laboratory specimens, and the authors agree with Mr. McAdam that the development of corrosion-fatigue pits is not appreciably influenced by polishing or roughening of the surface.

As Mr. McAdam has pointed out, corrosion-fatigue endurance limits should apply only to service conditions closely approximating the laboratory testing conditions under which the tests were carried out. Since the paper was written, all corrosion-fatigue S-N curves of these five steels have been carried out to 100 million cycles of stress without changing the values reported in Table V. One solid specimen of steel D (see *S-N* diagram, Fig. 3) at a stress of 14,000 lb. per sq. in. was run to 399 million cycles of stress without fracture.

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[7] Associate, and Assistant Professor, respectively, Department of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[8] H. F. Moore and J. B. Kommers, "An Investigation of the Fatigue of Metals," *Bulletin No. 124*, Engineering Experiment Station, University of Illinois (1921).

The additional data presented by Mr. Harder give an interesting comparison of relative life of several steels under constant maximum stresses in conditions closely simulating a particular service condition. However, there is always a question as to whether these materials might possibly be arranged in a different order of relative life if compared on the basis of tests at a higher or a lower stress.

TABLE I.—AXIAL FATIGUE PROPERTIES.

Material	Endurance Limit in Repeated Tension,[a] lb. per sq. in.	Yield Point, lb. per sq. in.	Tensile Strength, lb. per sq. in.
A........	60 000	60 600	78 400
B........	60 000	56 000	70 800
C........	83 000	63 000	91 300
D........	84 000	81 300	125 000
36........	46 000	36 500	57 400

[a] Based on 15 million cycles of stress.

Since presentation of the original paper, additional data have been obtained on the endurance limits of the five steels when subjected to repeated tensile stresses in air, the stress in each cycle varying in magnitude from zero to maximum. These values are listed in the accompanying Table I.

It should be noted that in all cases the endurance limits based on 15 million cycles of stress are about equal to or greater than the yield points of the materials. Consequently design stresses for actual service conditions similar to the test conditions probably should be based on the yield points of the materials rather than on the endurance limits.

In connection with Table V of the paper, supplementary data on steel 36 for specimens with stress raisers are now available. The endurance limits of steel 36 for specimens with a fillet are 17,000 lb. per sq. in. in air, and 16,000 lb. per sq. in. when tested in water; for specimens with a hole the endurance limits are 16,000 lb. per sq. in. in air, and 14,000 lb. per sq. in. when tested in water. The structural carbon steel thus has about the same strength under these corrosion-fatigue conditions as the low-alloy steels that were tested.

ACTUAL GRAIN SIZE RELATED TO CREEP STRENGTH OF STEELS AT ELEVATED TEMPERATURES

By S. H. Weaver[1]

Synopsis

A statistical analysis is made of 32 creep tests which have in common the chemical composition of the steel (S.A.E. No. 4330 steel) and a creep temperature of 450 C. (840 F.). Correlation of the creep strength with the actual size of the existing carbide grain shows that there is an optimum grain size for the maximum creep strength; a larger or smaller grain than the optimum value gives a decreasing creep strength.

For this particular steel and temperature

(*a*) Changing the grain size can vary the creep strength by 21,000 lb. per sq. in.

(*b*) The structure within the grain can change the creep strength by 4500 lb. per sq. in.

(*c*) Ferritic banding in the visible mechanical form can lower the creep strength up to 4300 lb. per sq. in.; while in the "masked" or distributed form the effect of banding is negligible.

In the creep tests made by the General Electric Co. there is a collection of 32 tests all made at the same temperature of 450 C. (840 F.) and all on the same steel with a chemical analysis within the commercial limits of what is now known as S.A.E. No. 4330, a nickel-chromium-molybdenum steel. These creep tests were made in the same laboratory with the same equipment, methods and organization. With these conditions common to all the tests, the creep strength for a constant creep rate of 1 per cent per 100,000 hr. differs for each test item as shown in Fig. 1 where the weakest steel is rated at 10,800 and the strongest steel at 40,200 lb. per sq. in. The secret of this four to one variation in creep strength must therefore lie within the tested metals.

The data with the samples of tested steels have been the subject of intensive study for some time past by the members of the Creep Committee of the General Electric Co. Some basic observations were made. Wyman[2] found that the weaker steels had dendritic segregations known as ferritic banding. G. H. Wright found that the stronger steels were obtained by heat treating at 950 C. (1740 F.) and holding at that temperature for at least 8 hr. before cooling and tempering. For a correlation study of the test data the above observations lead to the first subdivision of the tests into (a) uniform and (b) non-uniform microstructures of the tested steels. This classification is shown in Fig. 1.

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[1] Turbine Engineering Dept., General Electric Co., Schenectady, N. Y.

[2] L. L. Wyman, "The Creep of Steels as Influenced by Microstructure," *Mechanical Engineering*, Vol. 57, No. 10, October, 1935, p. 625.

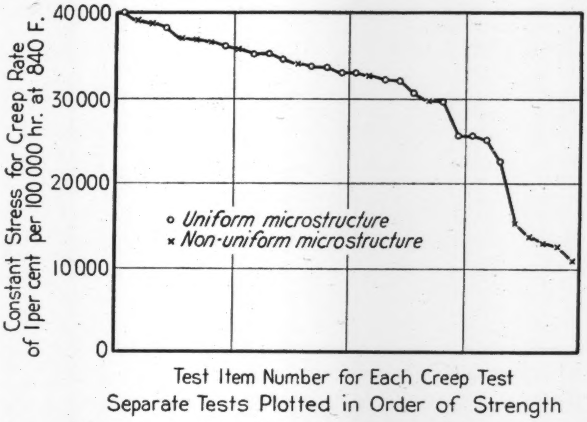

FIG. 1.—Variation in Creep Strength for Nickel-Chromium-Molybdenum Steels with Same Chemical Analysis and Temperature.

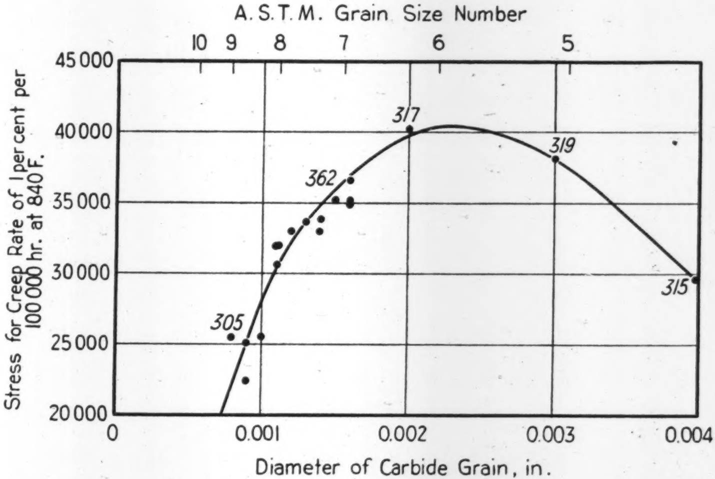

FIG. 2.—Creep Strength of Nickel-Chromium-Molybdenum Steel, S.A.E. No. 4330 at 450 C. (840 F.)

Grain Size:

Consider the group of creep tests which have a uniform microstructure in the steels. Neglecting the small differences in chemical analysis, the test items should differ in (a) the grain size, (b) the metallurgical structure within the grains and (c) the proportion of ferrite grains to carbide grains in each test piece. Fortunately, this subgroup of tests had no separate grains of ferrite and all of the grains are of a similar sorbitic structure so that the only variable is the size of similar grains.

The grains considered here are the actual grains that exist in the steel during the creep extension. The size is determined by actual grain boundaries and is the average diameter of the carbide grain read from a photomicrograph at a

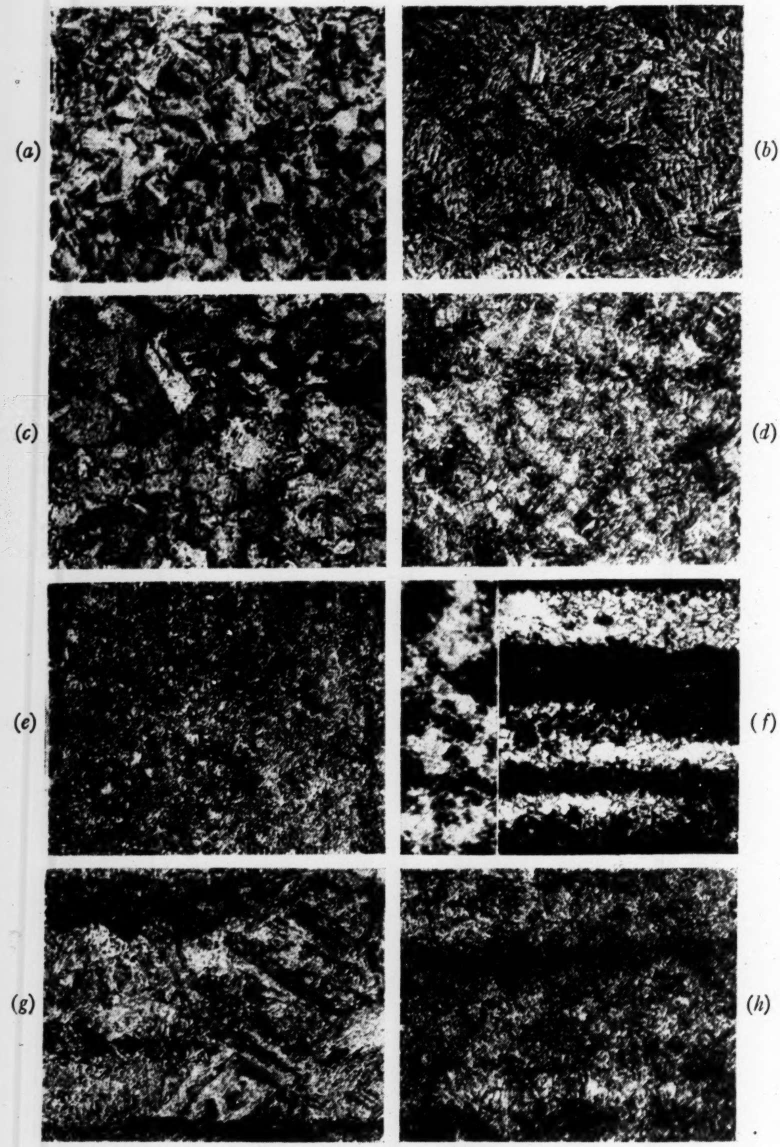

FIG. 3.—S.A.E. No. 4330 Steel at 450 C. (840 F.) (× 100).

	ITEM NUMBER	CREEP STRESS[a]	GRAIN DIAMETER, IN.		ITEM NUMBER	CREEP STRESS[a]	GRAIN DIAMETER, IN.
(a)	315	29 700	0.0041	(e)	305	25 600	0.0008
(b)	319	38 200	0.0030	(f)	609	10 800	0.0007
(c)	317	40 200	0.0020	(g)	358	39 100	0.0024
(d)	362	36 500	0.0016	(h)	365	32 700	0.0016

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[a] Creep stress, pounds per square inch, for creep rate of 1 per cent in 100,000 hr.

magnification of 100. The 18 tests with uniform microstructures in the steel have the creep strength plotted against average grain diameter in Fig. 2. The curve shows an optimum grain size to obtain the maximum creep strength for this steel and temperature. The crest of the curve is rather broad and includes tests with heat treatments ranging from oil quenched and tempered, normalized and drawn, to an anneal. These heat treatments for the greater creep strengths involve a grain growth at a higher treating temperature and holding at that temperature for a longer than usual time.

There can be a question about the larger grained item 315 at the end of the curve in Fig. 2. This steel was slow cooled at a rate of 30 deg. Cent. per hr. from 950 C. (1740 F.) with the structure shown in Fig. 3 (*a*). Repeated trials on the microstructure developed only an occasional grain boundary. The omission of this test point would not change the shape of the curve in Fig. 2.

Figure 3 (*b*) covers item 319 which was air cooled after holding at 950 C. (1740 F.) for 8 hr. followed by a draw at 650 C. (1200 F.) held 1 hr. and air cooled.

Figure 3 (*c*) gives the structure of item 317 which had the same heat treatment as Fig. 3 (*b*).

Figure 3 (*d*) shows the oil quenched structure of item 362 which was held at 925 C. (1695 F.) for 8 hr., oil quenched, then followed by a temper at 575 C. (1065 F.) for 3 hr. and air cooled.

Figure 3 (*e*) for item 305 goes to the small-grained end of the curve in Fig. 2 where the heat treatment was 850 C. (1560 F.), held for 1 hr. and oil quenched followed by a temper at 650 C. (1200 F.) for 1 hr. and cooled.

These steels were obtained from many sources—usually commercial—so that their grain growth characteristics differ. There is a general relation between the grain size and the temperature and time in heat treatment which can be traced as one travels along the curve in Fig. 2. Beginning at the fine-grained or origin end of the curve the first three points have 825 to 850 C. (1515 to 1560 F.), 1 hr., oil quench and temper; the next cluster of tests increase from 850 to 925 C. (1560 to 1695 F.) for 1 hr.; then 850 C. (1560 F.) for 4 hr. to 925 C. (1695 F.) for 8 hr. with oil quench and draw; then begins the numbered test points with 950 C. (1740 F.) for 8 hr. but with different rates of cooling. The general trend of these heat treatments follow the known characteristics of steel for increase in grain size.

The present objective is to establish an optimum grain size for maximum creep strength at elevated temperature from a group of tested steel structures which differ only in the actual size of the grain. The numerical values in the conclusion are necessarily limited to this type of steel and temperature of creep test.

Banded Steel:

Consider the group of creep tests which have a non-uniform microstructure in the steels. Each test is represented by a cross in Fig. 1. In all cases the non-uniformity in structure is due to ferritic banding in rolled steel.

An extreme but not unusual example of banding in this grade of rolled steel is given in Fig. 3 (*f*) for test item 609 which presents the microstructure of the 10,800 lb. per sq. in. creep strength steel, the lowest value in Fig. 1. The heat treatment is an anneal at 825 C. (1515 F.) held 1 hr. and furnace cooled. The right side of Fig. 3 (*f*) is a section parallel with the axis of the bar and shows the small grains and the continuous strings of (white) ferrite (iron and nickel) running lengthwise of the rolled bar. The left side of Fig. 3 (*f*) is a cross-section at right angles to the length of the bar and shows the cross-section of the continuous strings of ferrite. From the mechanical form of the bands a fair estimate can be made of the effect upon the creep strength of the bar. The cross-section of the ferritic strings cover 43 per cent of the bar area. If these white strands had zero strength the dark metal would carry the total load at 18,950 lb. per sq. in. stress. But the ferrite has a creep strength, about one-third that of the alloyed steel, which would place the stress in the white metal at 5000 lb. per sq. in., the stress in the dark metal at 15,100 lb. per sq. in. against the total bar creep stress of 10,800 lb. per sq. in. This difference in the creep strength of about 4300 lb. per sq. in. is the maximum that can be charged against the presence of the mechanical form of ferritic banding in this class of steel at 450 C. (840 F.) creep temperature.

However, heat treatments can change not only the mechanical form of the banding but produce different size of grains and metallurgical structures within the grains.

Figure 3 (*g*) for test item 358 shows the grain size and structure for the 39,100 lb. per sq. in. creep strength test, the strongest steel with a non-uniform structure in Fig. 1. The treatment was 950 C. (1740 F.) held for 8 hr., air cooled and followed with a draw at 625 C. (1155 F.) for 3 hr. and air cooled.

Figure 3 (*h*) for test item 365 shows the effect of an oil quench and high draw temperature. This steel was treated at 925 C. (1695 F.) held for 8 hr., oil quenched and followed by a draw at 650 C. (1200 F.) for 3 hr. and air cooled The result is an intermediate grain diameter (0.0016 in.) and creep strength (32,700 lb. per sq. in.).

The mechanical form of the banding due to the anneal heat treatment of the steel in Fig. 3 (*f*) is "masked" by the faster cooling used in the heat treatments for the steels in Figs. 3 (*g*) and (*h*). The weakness caused by the mechanical form of the bands is eliminated. The higher temperatures and longer holding time used in the latter heat treatments result in a larger grain size, while the more rapid cooling rates produce sorbitic (gray) structures within the grains.

The group of 14 tests with non-uniform microstructure contain at least three active variables: (*a*) ferritic bands, (*b*) grain size and (*c*) structure within the grains; a fourth variable is the proportion in area of ferrite grains to carbide grains but this occurs only in the annealed steels with the mechanical form of bands and, for the present, this factor can be combined with variable (*a*). With the three variables active, no correlation of test values could be formed. When the data are plotted on Fig. 2 the test points scatter over the area included between the curve and a 10,800 lb. per sq. in. horizontal line.

With the two principles previously discussed, that of the influence of size of grain and that of the effect from the mechanical form of bands, one can make some interesting but speculative deductions from the above test results. A respresentative example is to make the weakest tested steel shown in Fig. 3 (*f*) into the strongest tested steel shown in Fig. 3 (*g*). This procedure is permissible because Fig. 3 (*f*) is reversible with Fig. 3 (*g*) and (*h*) by re-treating with the respective heat treatments. To diffuse the ferrite out of the mechanical form in Fig. 3 (*f*) requires 1250 C. (2280 F.) for 5 to 10 hr. The lower heat treating temperatures used produce a negligible diffusion not affecting the reversibility. The variation in the following estimates is due to the assumption that all test bars compared have 43 per cent of section area in ferrite segregation. Actually, this percentage varies somewhat.

Compare the tests for Fig. 3 (*f*) with Fig. 3 (*g*). Figure 3 (*f*) had a tested creep strength of 10,800 lb. per sq. in. It was previously estimated that if the mechanical form of the continuous bands was absent the alloyed steel would have its creep strength increased to about 15,100 lb. per sq. in. If the grain size is then changed from 0.0007 to 0.0024 in., from the curve in Fig. 2, the creep strength is increased by 21,000 lb. per sq. in. stress which brings the bar up to 36,100 lb. per sq. in. creep strength. The third variable is the change in microstructure within the grain. In this case an imperfect lamellar pearlite changes to an acicular sorbite, both characteristic of this type of steel. The effect of this change in structure is measured by the difference between 36,100 lb. per sq. in. and the tested creep strength of Fig. 3 (*g*) which is 39,100 lb. per sq. in. or a change of 3000 lb. per sq. in. in the stress. By similar estimates from the different tests the influence of the three variable factors in creep may be stated in the following approximate stress values for S.A.E. No. 4330 steel at 450 C. (840 F.).

(*a*) With ferritic banding present in the mechanical form that is produced by an anneal heat treatment the effect on the creep strength depends upon the percentage of cross-sectional area occupied by bands; 43 per cent is the largest found in this series of tests. The amount of banding present in this group of tests can produce a decrease in creep stress from 0 to 4300 lb. per sq. in.

(*b*) The size of the existing carbide grain in the steel from Fig. 2 can independently change the creep stress by a maximum of 21,000 lb. per sq. in.

(*c*) From estimates similar to the above example, the change in metallurgical structure within the grains can influence the creep stress from 0 to 4500 lb. per sq. in.

The above ratings in creep stress gage the relative importance of three factors which enter into the creep strength of S.A.E. No. 4330 steel at 450 C. (840 F.). The influence of grain size predominates and ranks in importance with that of alloy additions to a carbon steel.

This correlation of creep data is concerned solely with the actual physical aspect of the polycrystalline metal which existed during the creep test. Detailed differences in the manufacturing process of the same type of steel give different grain growth temperature characteristics; therefore duplicating the details of a heat treatment on a different heat of steel does not always produce the same creep strength.

DISCUSSION

MR. SABIN CROCKER[1] (*presented in written form*).—By way of prefacing his remarks the writer begs leave to discuss this paper from the viewpoint of a mechanical engineer seeking advice from metallurgists in selecting materials for high-temperature steam piping. At the present time power plant designers hesitate to employ steam temperatures above 925 to 950 F. because of uncertainty regarding the behavior of materials at higher temperatures. By the same token, designers may be using heavier walled pipe and superheater tubes for present temperatures than would be needed if they knew what to specify by way of placing available material in the most suitable metallurgical condition.

The fuel saving attainable through going to higher temperatures, either with or without the higher pressures made feasible thereby, would be considerable, and each year's delay in reaching the highest practicable operating temperature for a given material is perpetually adding that much to the fuel bill of all plants built during the interval. Is it not possible that part of this delay is avoidable and due to lack of a full understanding between metallurgists and designing engineers? In other words, is there not a chance that what metallurgists discover this year does not influence engineering design until next year or the year after? Without wishing to be thought unduly critical the writer would like to point out the possibility for metallurgists getting their ideas across to engineers in a form more understandable to the latter.

For one thing it often happens that the results of creep investigations are reported in terms which, while wholly intelligible to the investigators are extremely bewildering to others. The writer doubts whether metallurgists realize how baffling their methods of expression are at best to those not sufficiently versed in the art, and wishes to make this plea for striving to achieve clarity through adherence to standard terminology which can be interpreted through reference to a recognized text, supported by adequate explanation of new terms.

This part of the discussion is not directed at this paper in particular since much of the confusion arises from the tendency for different authors to use the same term to convey different meanings, or on the other hand to have too many different names for the same thing. Without exaggeration the writer can assure you that he has known eminent metallurgists to be at a loss in understanding the true significance of terms used by equally eminent men of the same profession. Confusion is bound to exist unless this situation is clarified by better definition of metallurgical terminology coupled with more adequate explanation of doubtful meanings. Until this is done, designing engineers will continue missing the full import of much that might be of value to them in such papers.

Since the present subject of *grain size* is typical of the metallurgical terms which baffle full understanding, it suggests the need for adequate definition of the various sorts of "grain size" under consideration in this or similar connection, and the relation if any between these various kinds of grain size.

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[1] Senior Engineer, Engineering Division, The Detroit Edison Co., Detroit, Mich.

The following statement appears on page 2 of the present paper: "The grains considered here are the actual grains that exist in the steel during the creep extension. The size is determined by actual grain boundaries and is the average diameter of the carbide grain read from a photomicrograph at a magnification of 100." The writer has no particular criticism of this statement aside from calling attention to the fact that the author does not define what he means by "carbide grain" in this connection and the writer cannot find it defined for a hypo-eutectoid steel in such texts as are available to him. This, however, is not the only grain size which investigators are coupling with creep strength.

In the current Report of the Joint Research Committee on Effect of Temperature on the Properties of Metals,[2] appears the following statement:

"In discussions of grain size in this report, unless specifically described otherwise, all grain size determinations are austenitic grain sizes resulting from the combination of times and temperatures used in the various heat treatments.

"These austenitic grain sizes are not to be confused with the McQuaid-Ehn grain size, which is the grain size obtained as the result of carburizing for 8 hr. at a temperature of 1700 F. and which may have no bearing whatever on actual grain size.

"The austenitic grain sizes listed in Table II are the actual austenite grain sizes established in the final heat treatment of the various materials prior to the creep tests, impact tests, and tension tests."

The following kinds of grain size have been encountered so far in this discussion:

1. Actual grain size,
2. Actual carbide grain size,
3. Actual austenite grain size,
4. McQuaid-Ehn grain size, sometimes referred to as inherent grain size.

Various investigators have linked creep strength to respective grain size of all four systems listed above. It seems pertinent, therefore, to ask which of the four sorts of grain size is most significant with respect to creep strength and whether there is some interrelation between them. The writer suggests this as a subject worth further investigation and analysis.

As having a possible bearing on establishing such a relation, attention is called to the following statement by Sauveur.[3] ". . . if the metal be kept a long time above its critical range, even but slightly above it, a coarser austenite will be formed, which in turn implies, after slow cooling, a coarser pearlitic or sorbitic structure. In hypo-eutectoid steel the grains of austenite will expel some free ferrite, and in hyper-eutectoid steel some free cementite, before being converted into pearlite, but the final pearlite grains will nevertheless increase in size with the size of the original austenite grains. The structure of steel containing 0.50 per cent carbon kept 2 hr. at 1150 C. and cooled in air has been shown in Fig. 182. The very large sorbitic grains formed prove the existence above the range of equally large, or even larger, austenitic grains. The cooling through the range was so rapid that but a small amount of free ferrite was separated, the sorbite grains, therefore, representing nearly the exact size of the austenite grains."

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[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part I, p. 153 (1938).

[3] Albert Sauveur, "The Metallography and Heat Treatment of Iron and Steel," Fourth Edition, p. 226, McGraw-Hill Book Co., New York City (1935).

In the light of what already has been said about the need for making new ideas on metallurgy available to plant designers as soon as possible, the writer hopes he will be pardoned for taking the liberty of pointing out in particular wherein he feels Mr. Weaver's paper could have been improved in this respect.

Supplementing, as it does, the present report of the Joint High Temperature Committee, this paper gives added emphasis to the influence of grain size on creep strength and the benefits of special heat treatments. While the difference in creep strength between coarse-grained and fine-grained steels has been considered at length by creep investigators since Kanter and Spring's work[4] on cast and forged materials, the differences directly attributed to grain size variations have not in general been of the magnitude found by the author for this particular steel or for the steels reported on by the Joint High Temperature Committee.

The writer has gained the impression that the work reported in the present paper must constitute but a small fraction of the data available to the author; otherwise the generality of the conclusions is entirely out of proportion to the test findings reported. Had the author chosen a more common steel his results not only would have been directly usable by more engineers, but the conclusions could have been substantiated to better advantage by other creep investigators from results of tests already made. While the S.A.E. No. 4330 steel used in these tests is essentially the same as the B4, C4, and F4 steels proposed for high temperature service by Subcommittee XXII of the Society's Committee A-1 on Steel, and closely resembles alloy steels used by the British Thompson Houston Co. in the 1000 F. turbine for Delray, it has not been used so extensively for 850 to 950 F. as has 0.5 per cent molybdenum steel.

In view of the present tendency for the central station industry to use carbon-molybdenum castings for valve bodies and turbine casings and carbon-molybdenum rolled material for pipe at service temperatures of 900 to 950 F., the author's findings as to the optimum grain size for that material and temperature would seem to be of more general interest in so far as superheated steam piping is concerned than the material chosen for demonstration. If similar test data for carbon-molybdenum steels at 925 F. are available, it is hoped the author will supply them. It would be most helpful also to learn what heat treatment the author would propose giving that material to produce optimum creep strength for these operating temperatures and the grain size corresponding thereto.

Only after diligent study of the previous paper by Weaver,[5] an article by Clark and Robinson,[6] and the treatise by Wyman[7] has the writer been able to find the exact composition of one of the specimens and the probable range of compositions involved. At least three of the specimens are of commercially rolled material. The creep test of the banded specimen, item 609, was of 3300-hr. duration and of the down-step variety. Presumably all the materials were of closely similar composition, of commercially rolled material, and all were tested by the down-step method so that the results are truly comparable. But such information should have been included in this paper.

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[4] J. J. Kanter and L. W. Spring, "'Long-Time' or 'Flow' Tests of Carbon Steels at Various Temperatures with Particular Reference to Stresses Below the Proportional Limit," *Proceedings*, Am. Soc. Testing Mats., Vol. 28, Part II, p. 80 (1928).

[5] S. H. Weaver, "The Creep Curve and Stability of Steels at Constant Stress and Temperature," *Transactions*, Am. Soc. Mechanical Engrs., November, 1936, RP58-16.

[6] P. H. Clark and E. L. Robinson, "An Automatic Creep Test Furnace-Guide," *Metals and Alloys*, February, 1935, p. 46.

[7] L. L. Wyman, "The Creep of Steels as Influenced by Microstructure," *Mechanical Engineering*, Vol. 57, No. 10, October, 1935, p. 625.

The attempt to correlate creep strength with actual grain size would be more convincing if the grain size were readily discernible on the micrographs presented in the paper. Question is raised as to whether this is a characteristic of the particular steel tested or whether the actual grain size on the micrographs could not have been brought out to better advantage. On the basis of the evidence presented in this paper, the efforts of the Joint High Temperature Committee[8] to relate austenitic grain size with creep strength appear to be more promising than the attempt to relate actual grain size. However, since both of these approaches to the problem of improving creep strength involve heat treatments at temperatures producing grain growth of the austenite, it is probable that both will be instrumental in producing steels with better creep resistance.

On first glance one is impressed with the demonstration that it could be possible to so heat treat a steel with a creep strength of 10,800 lb. per sq. in. that it would support a stress of 39,100 lb. per sq. in. with the same rate of extension. It is decidedly disappointing, however, to find on more careful reading that the demonstration is purely hypothetical and the writer is inclined to question whether a matter of conjecture should be accorded the weight given it under the guise of "speculative deductions."

Without wishing to detract in any sense from the valuable contributions to knowledge of the influence of grain size on creep strength afforded in the present paper, the writer wishes to express the hope that Mr. Weaver or his associates will present the further information suggested at the earliest opportunity.

MESSRS. A. E. WHITE[9] and C. L. CLARK[10] (*presented in written form*).—Mr. Weaver's paper gives additional information and data with respect to the creep strength of steels. It throws further light on the properties of nickel-chromium-molybdenum steels of the S.A.E. No. 4330 type at 840 F. in various conditions of heat treatment.

For some time, it has been appreciated that the high-temperature characteristics of steel are dependent upon many variables such as chemical composition, melting practice, ingot size, degree of reduction in hot working, heat treatment, uniformity of structure and grain size. From time to time, the emphasis given to these variables changes, and at present grain size appears to be receiving more attention than any of the others.

Much confusion has resulted over the matter of grain size. To some it may imply the so-called inherent grain size in steel as revealed by the McQuaid-Ehn test while others consider the grain size in terms of actual grain size—that is, the grain size which is visible after polishing and etching the steel in the condition in which it is to be used. In this paper, Mr. Weaver appears to consider the actual grain size based on the carbide constituent.

The work reported was done on steels of the S.A.E. No. 4330 type but the actual analysis of each heat considered is not given. The permissible limits for this type of steel are:

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[8] Progress Report on Study of Effects of Manufacturing Variables on the Creep Resistance of Steels (Project No. 18), Appendix V, Report of Joint Research Committee on Effect of Temperature on the Properties of Metals, *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part I, p. 149 (1938).

[9] Director of Department of Engineering Research, and Professor of Metallurgical Engineering, University of Michigan, Ann Arbor, Mich.

[10] Research Engineer, Department of Engineering Research, University of Michigan, Ann Arbor, Mich.

Carbon	0.25 to 0.35 per cent
Manganese	0.50 to 0.80 per cent
Chromium	0.50 to 0.80 per cent
Nickel	1.50 to 2.00 per cent
Molybdenum	0.30 to 0.40 per cent

It is quite conceivable that considerable differences in properties might be secured if one steel was on the high side with respect to carbon, manganese, chromium and molybdenum, and another steel on the low side with respect to these elements.

It is stated: "These steels were obtained from many sources—usually commercial . . . ." This raises the question as to whether all the tests were made on commercial heats, whether some were open-hearth and others electric-furnace steel, whether they were made on cast steel, forged steel, or rolled steel and whether, in the case of the rolled bars, appreciable differences existed in the degree of hot reduction.

It would be of assistance in evaluating the data if the author could supply this information, especially the analysis of each of the test specimens used, the steel melting process and practice employed, the initial size of the stock as cast, the amount and conditions of reduction, and the preheat treatment, if any. The inclusion of the room temperature tensile properties would also be of interest.

Assuming, however, that the material is all of substantially the same composition and was made and fabricated in substantially the same manner, a careful analysis of the data submitted in Fig. 2 should be made. It is questionable if all the differences in this figure can be laid to carbide grain size exclusively. This statement is made because we are called upon to compare the carbides in steels which have been oil quenched and drawn with the carbides in normalized and drawn steels and the carbides in annealed steels. It is questionable if the carbides resulting from these three different types of heat treatment, or the matrix in which these carbides are dispersed, are similar in all respects except for the carbide grain size. In fact, the findings submitted in this figure are quite in line with work reported by us several years ago in which it was shown that a normalized-and-drawn structure possessed superior creep resistance to either an oil-quenched-and-drawn or an annealed structure.

The one real comparison which can be made on the basis of carbide grain size is in the case of the oil-quenched-and-drawn steels. The carbides in these steels are presumably all of the same type and therefore, the one variable, assuming the steels were identical in other respects, is that of carbide grain size. These show that as carbide grain size increases there is an increase in the creep strength.

In Fig. 3, it is difficult to understand how samples (*b*) and (*c*), presumably of the same type, can have had the same heat treatment without the existence of variables which have not been reported. The structures appear to be radically different and the difference is not confined to carbide size alone.

The work done on the banded steel is most interesting but it is not believed that the conclusions drawn with respect to the magnitude of the influence of banding on the resulting creep resistance are wholly convincing. There is no question but that banding will decrease the creep resistance of steels of this type; but there are questions as to the magnitude of this decrease. The author's results definitely show the banded steel to have the lowest creep resistance of the steels considered. The banded steel was, however, given a different heat treatment than any of the remaining steels considered in Fig. 3 and, in fact, the temperature used in annealing was lower than that employed in any of the other heat treatments. This may be an important contributing factor and in order definitely to indicate the influence of banding, two steels, as nearly identical as possible and in the same condition of heat treatment but which differ only in the degree of banding present, should be considered.

The changes in creep stress values which the author has given in the summation and elsewhere in his paper are doubtless intended to be used with discretion. They are based largely on assumptions and should be proved experimentally before they can be taken too literally. For example, in the case of the banded steel it is assumed that the ferrite has a creep strength of about 5000 lb. per sq. in. This value is open to question as the ferrite in a steel of this type would contain certain of the alloying elements in solid solution and this, as well as many other factors, might influence the strength of ferrite. Likewise, while it is agreed that the size of the existing carbide grain in steel will influence the creep strength, it is believed that in attributing the entire range of 21,000 lb. to carbide grain size exclusively may be open to question.

It should be pointed out that this work was done at 840 F. and such conclusions as are drawn should be on the basis of this temperature. It is known that different steels manifest different characteristic tendencies at different temperatures. One can never be assured how they will act at other temperatures until after test. Also, factors which affect the creep properties of a steel at a given temperature in one direction may work in a reverse direction at other temperatures. Therefore, one must not draw conclusions too hastily and assume that the results presented are indicative of what would take place at all other temperatures. This is a general comment; it must not be construed as a criticism of any of the statements made by the author of the paper as he confined his paper to findings at 840 F.

The author is to be congratulated upon the material covered and presented in the paper. The information is somewhat disquieting to those who would like to see steel of the same chemical composition possess the same physical properties.

Possibly the one hopeful feature is that the sooner we more fully realize the influence of variables on creep properties, the sooner we may be able to control these variables so as to get metal for any given temperature which has a relatively uniform creep rate at any given temperature.

MR. H. W. GILLETT[11] (*presented in written form*).—This paper is a peculiar combination of interesting and valuable information, and possible misinformation the latter being contained in Fig. 2. There is grave danger that a casual reader will look at and remember Fig. 2 without careful study of the text. It purports to plot grain size against creep rate, but there are, as the author points out in the text, several other factors, and assurance is lacking that all extraneous factors have been eliminated. The conclusion that creep resistance is at a maximum at grain size 6, is not consistent with other information nor with a logical process of reasoning.

In view of the author's own statement that sample 315 really has an unknown grain size, it would appear that if the draftsman had read the paper which Fig. 2 was to accompany, and were reasonably conscientious, he would have dotted the descending branch of the curve.

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[11] Metallurgist, Battelle Memorial Inst., Columbus, Ohio.

The grain size terminology used is confusing. The term "actual" grain size is often used to signify that the austenitic grain size established by the heat treatment used is given rather than the McQuaid-Ehn grain size found by carburizing 8 hr. at 1700 F. Just what is meant by "carbide" grain size is not at all clear. The grain size that most investigators have compared with creep results is the austenitic grain size. The author has apparently attempted to evaluate this or some undescribed combination of this and something else from the specimens as heat treated. It is well known to metallurgists that it is extremely difficult to tell from the final microstructure what the austenite grain size was, so use is made of the fact that the grain size established by time and temperature in the austenite is not altered by reasonable variations in the subsequent cooling rate; so grain size is determined by regulated cooling or quenching, and then etching in such fashion that the grains are delineated. A specimen heated just as is the test specimen, but differently cooled, is sacrificed in order to get reliable information when the regular heat treatment yields such doubtful data as appear in this case. That this is expected to be done in creep testing is indicated by looking at the regular form for making a creep report,[12] where the austenitic grain size is to be stated. In the A.S.M. Handbook[13] rather specific directions are given as to the method of determining austenite grain size in carbon and alloy steels.

If specimens of the 32 steels had been given the same time and temperature of heating that the creep specimens had had, and the austenitic grain size had been determined on them, the haziness of this paper would have been dispelled and usable information obtained through the grain size correlation. Without this, the paper at least contains an aching void and very possibly draws conclusions that a more thorough study would materially modify. The job should be supplemented by further data on grain size, determined by the methods of the metallographer, rather than the astrologer.

MESSRS. R. F. MILLER[14] and R. H. ABORN[14] (*presented in written form*).—We who are running creep tests have often heard the complaint from design engineers that creep data are of little value because the results obtained for a given steel vary over wide limits. It has also been stated that high-temperature short-time tension tests render more useful information because the results are in closer agreement than are those of creep tests. Yet this attitude loses sight of the fact that creep tests are more sensitive and disclose actual differences which are not disclosed by the short-time tension test.

A few months ago we presented a paper before the American Society for Metals[15] showing the marked influence of heat treatment on the creep characteristics of two low-alloy steels. Mr. Weaver in his paper has presented evidence of even more striking variation in creep strength of different specimens of a single grade of steel, all nominally S.A.E. No. 4330. The results obtained under constant test conditions show that the stress required to produce a creep rate of 1 per cent per 100,000 hr. may vary from

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[12] Form for Reporting Data and Results of Long-Time (Creep) High-Temperature Tension Tests, Appendix I to Tentative Method of Test for Long-Time (Creep) High-Temperature Tension Tests of Metallic Materials (E 22 - 38 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part I (1938); also 1938 Book of A.S.T.M. Tentative Standards, p. 1668.

[13] E. C. Bain and J. R. Vilella, "Austenitic Grain Size in Steel," *Metals Handbook*, 1936 Edition, Am. Soc. Metals, p. 593.

[14] Research Laboratory, U. S. Steel Corp., Kearny, N. J.

[15] R. F. Miller, R. F. Campbell, R. H. Aborn and E. C. Wright, "Influence of Heat-Treatment on Creep of Carbon-Molybdenum and Chromium-Molybdenum-Silicon Steel," *Transactions*, Am. Soc. Metals, Vol. XXVI, No. 1, March, 1938, p. 81.

22,000 to 40,000 lb. per sq. in. at 840 F., as shown in Fig. 2. This variation is due primarily to differences in mode of heat treatment and may also be influenced by differences in manufacturing practice and composition. This paper should prove most illuminating to those who ask for a single figure for the creep strength of any grade of steel at any one temperature.

Without doubt grain size is one of the significant factors influencing the creep rate of a steel, but when it is brought in under the dubious title of "carbide grain size" with no more supporting evidence than is provided by the unsatisfactory photomicrographs in Fig. 3, one cannot but question the validity of the author's interpretation of his data according to which A.S.T.M. grain size No. 6 is the optimum.

When one writes down the corresponding heat treatment beside each plotted point in Fig. 2 it becomes obvious that the points represent a range of heating temperatures and times, followed by cooling rates ranging from furnace cooling to oil quenching and these in turn followed by differing tempering schedules. The resulting structures tested in creep will differ so much in type and degree of carbide dispersion, amount of "free" ferrite, etc., that an apparent grain size in one specimen may have little or no relation to that in another. If instead, one considers the austenite grain size established at the initial high temperature, the picture becomes much clearer. It is well known that austenite grain size tends to increase with temperature and with time at temperature. On the ascending branch of the curve in Fig. 2, the heating temperature rises from 825 to 950 C. and the time from 1 to 8 hr. Thus, it is reasonable to assume that the stress to produce a given creep rate rises with increasing austenite grain size—an assumption proved by recent work at Battelle Institute and reported at this meeting.

The descending portion of the curve in Fig. 2 is based on two tests. In one test (319) the heat treatment was identical with test 317 at the "peak" of the curve, and therefore one would expect a similar structure. Test 319 although reported to be coarser than the "optimum," actually appears from Fig. 3 (*b*) to be composed of units somewhat smaller than those in test 317 (Fig. 3 (*c*)) which would place it on the ascending branch of the curve. The second test (315) differed fundamentally from all the others in that it was slowly cooled and not reheated. Its properties, irrespective of any grain size rating, would be markedly different and the author, in fact, doubts the value of this point.

The probable influence of grain size reduces then to one salient trend: namely, that increasing the austenite grain size increases the resistance to creep. The fact that the highest resistance to creep was produced by a treatment consisting of 8 hr. at 950 C. followed by air cooling and reheating at 650 C. for 1 hr. does not preclude the possibility that a somewhat larger austenite grain size produced at a higher temperature might still further improve the resistance to creep. Without doubt, when one takes into account other mechanical properties such as notch toughness, which a steel must possess for most applications at elevated temperature, an optimum grain size does exist, although in a different sense from that in which Mr. Weaver uses the word.

It is to be hoped that the author will conduct further tests in which all factors, including manufacturing practice, are maintained constant, with austenite grain size as the sole variable.

Although this paper does not prove the existence of an optimum grain size for maximum creep strength, a possible justification for the existence of an optimum grain size, at least in a plain carbon steel, suggests itself. During our study of the effect of heat treatment on the creep resistance of carbon-molybdenum steel, we learned that structural change such as spheroidization during the creep test tended to accelerate the creep or flow of the metal. Furthermore, we know that the smaller the size of the carbide particles in the initial structure, the more rapidly they tend to spheroidize and coarsen. Also, the higher the temperature to which a steel is heated prior to creep testing, the coarser the austenite grain size and the finer the size of the particles in the Widmanstatten pattern formed during air cooling.

Thus, if a series of specimens was normalized from successively higher temperatures before creep testing, it might be found that the creep strength would be greater the coarser the austenite grain size up to a certain maximum size, and that further coarsening would weaken the steel because the carbide particle size in the Widmanstatten patterns had become so fine that its rate of spheroidization during the creep test would accelerate the flow enough to offset the effect of further coarsening of the austenite grain size.

In short, it is our belief that the creep strength of a given steel is associated not only with its grain size, but also with the mode of distribution of the carbides and the stability of this distribution during the test.

MR. P. E. MCKINNEY[16] (*presented in written form*).—In recent years the number of factors believed to influence the creep behavior of steels has been rapidly increasing, but in almost no case has the quantitative effect of these variables been established. It is, therefore, quite reassuring to note that Mr. Weaver has been able to attribute a consistent effect to one of the variables.

Knowing the grain size of the steel with which the author has dealt, there can be little doubt but that its creep strength at 840 F. can be accurately predicted. He has rightly said, therefore, that grain size is the controlling factor. Is it possible, however, that grain size is itself a manifestation of or index to a more fundamental attribute? We wonder, in other words, whether the property from which the steel derives its creep strength may not be the carbides which are present in a more or less submicroscopic form. It has been thought in connection with other high-temperature steels containing elements of the carbide forming type, that the particular carbides contained together with the condition in which such carbides exist, may be responsible for the fact that these steels offer greater resistance to creep than do steels in which the only carbide present is that of iron.

If it be true that carbides of chromium, molybdenum, and other elements are more stable than iron carbide at elevated temperatures, and can exist in a finely divided state without agglomeration or decomposition, it would seem possible that at a certain critical submicroscopic size they should offer maximum resistance to flow, due to a keying action in the slip planes of the steel. We would imagine that the size of carbide particle and size of grain are both entirely controlled by heat treatment and are themselves accordingly completely correlated. Thus, the grain size of the steel may simply indicate the size of the submicroscopic carbide particles. This reasoning is, to be sure, rather speculative and does in no way contradict the fact that in Mr. Weaver's series of tests grain size was the controlling factor.

Aside from the theoretical considerations of the author's paper, the test results constitute, to our mind, an excellent justification for the policy of most steel producers in refusing to publish minimum guaranteed creep strength values for the various high-temperature alloy steels. He has shown that the limiting creep stress obtainable for S.A.E. No. 4330 steel is different for almost every one of his 32 tests. While the policy of publishing data of this kind has been followed in the past by some organizations, it is gradually becoming a more general policy to refrain from broadcasting any creep information which might be construed as a guarantee of equal or better creep strength regardless of manufacturing or heat treatment operations. Mr. Weaver's paper is, in other words, a logical explanation for the different creep properties obtained on different heats of the same analysis. It is a definite contribution to the problem and should receive careful attention in connection with other grades of steel.

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[16] Metallurgical Engineer, Bethlehem Steel Co., Inc., Bethlehem, Pa.

While there are other variables which may enter the creep problem, such as method of steel manufacture, deoxidation, etc., I am hopeful that this investigation will hasten a satisfactory solution to the questions which still exist in connection with the differing creep behavior exhibited by various heats of steel of the same analysis.

MR. S. H. WEAVER[17] (*author's closure*). —A paper which was strictly limited to a statistical analysis of test results has brought forth some interesting discussions upon the causes which produced the characteristics indicated by that study. This is partly due to the novelty of the viewpoint expressed in the paper. Creep strength is considered a physical property of the steel dependent upon the conditions which exist within the metal during the plastic flow, and the creep properties can be judged by (1) the chemical composition and (2) the physical forms that actually exist within the microstructure of the metal during creep.

The steel maker and metallurgist are interested in the many variables that enter into the history of the steel. The importance of these manufacturing processes is realized. The steel manufacturer and the theoretical metallurgist meet on common ground by using the austenitic grain as the unit of measurement for the steels' characteristics. But the austenitic grain decomposes upon cooling through the transformation temperature into differently sized products for the differently alloyed steels and for different rates of cooling in the same steel. In the present tests the grain size in the steel existing after transformation and during the creep test is often but not always the same size as the austenitic grain. When a similar heat treatment is applied to a weldable alloy steel such as 0.20 per cent carbon, 0.50 per cent molybdenum, there results from a single austenitic grain a number of areas of ferrite and an area with precipitated carbon as carbides, all areas with well-defined boundaries giving mechanical and physical entities which exist during creep of the steel. These areas in the microstructure, here called existing grains, are the diameters used as the unit of measurement for the creep properties and not the non-existing austenitic grain with its variable products. While the manufacturing history of the steels and the austenitic grain size were intentionally omitted in the paper as not pertinent to the statistical analysis of test results, each discusser has made a reference to them. The recorded data follows:

The microstructures of the principal test items in Fig. 2 of the paper are reproduced at 1000 diameters from the same specimens previously used. The

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[17] Turbine Engineering Dept., General Electric Co., Schenectady, N. Y.

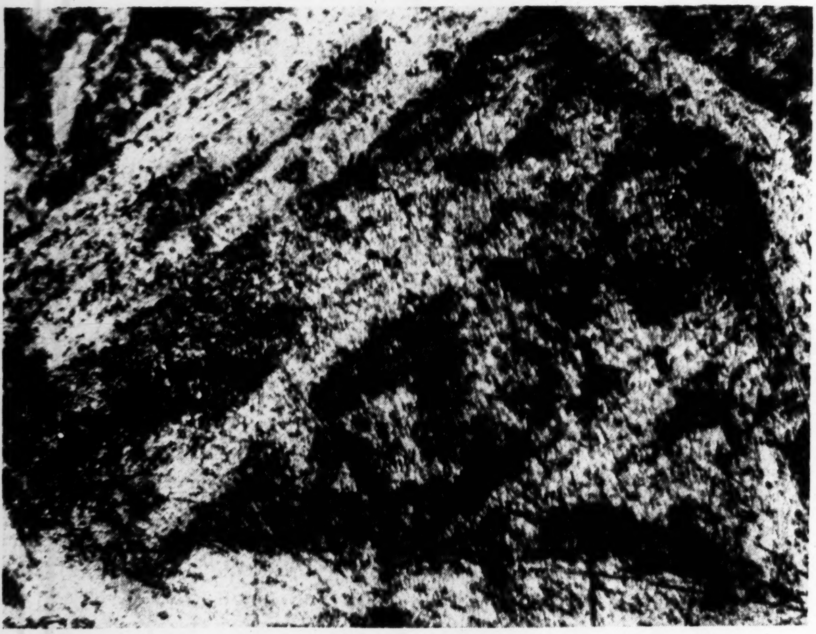

FIG. 1.—Item 315 (× 1000).

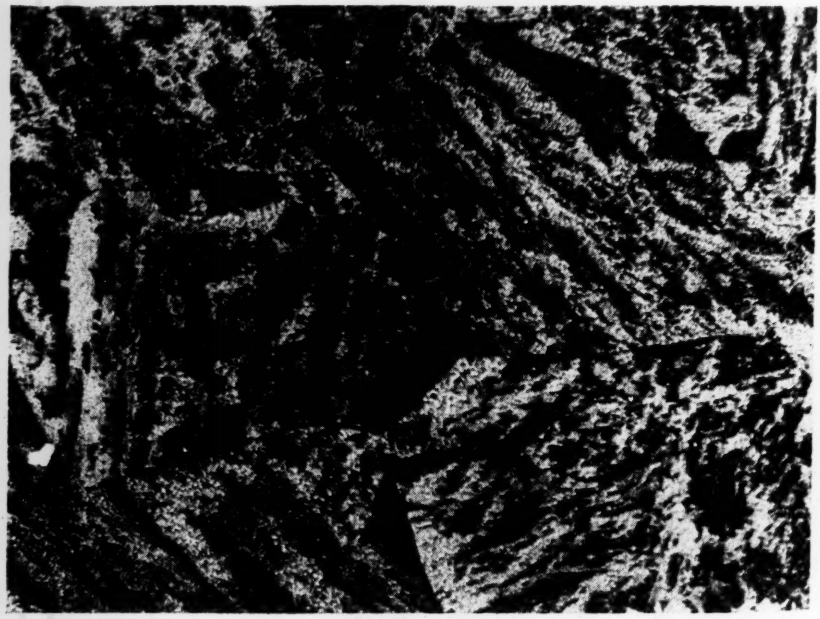

FIG. 2.—Item 319 (× 1000).

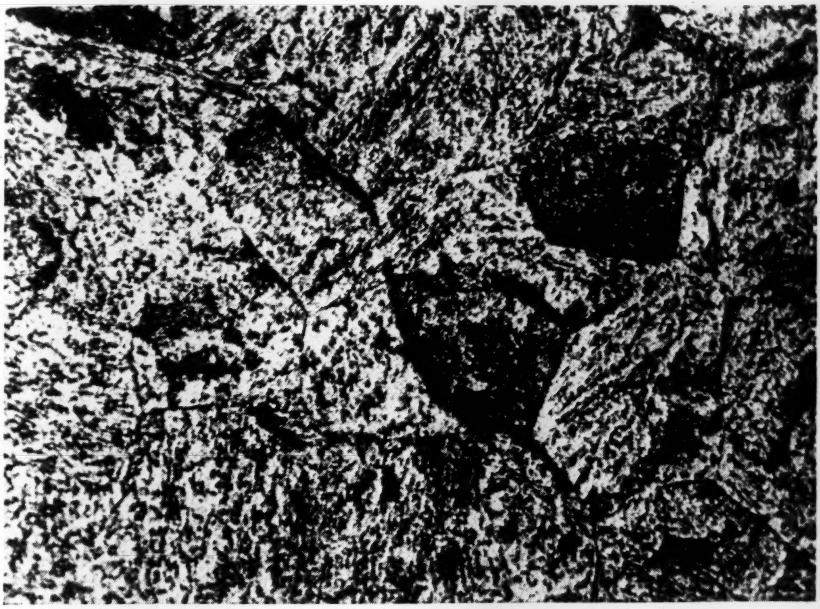

FIG. 3.—Item 317 (× 1000).

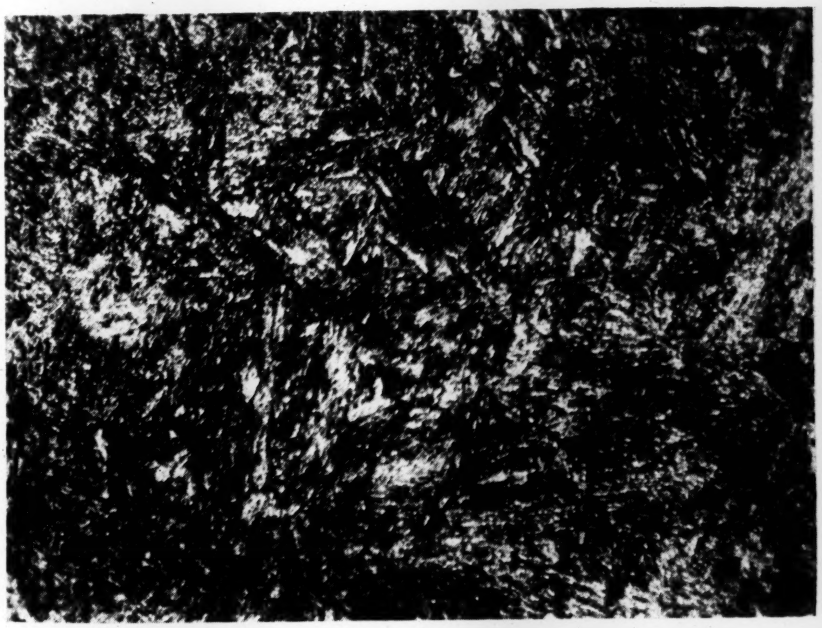

FIG. 4.—Item 362 (× 1000).

carbide formations within the grains were classified as sorbitic.

The accompanying Fig. 1 is item 315. At the slow cooling rate of 30 deg. Cent. per hr. the steel is so sluggish that the austenitic grain during the transformation only separated enough ferrite to outline dimly the grain boundary. The prior austenitic grain and the existing grain are practically the same size. The structure within this coarse grained annealed steel was classified as sorbitic.

The accompanying Fig. 2 is item 319, cooled in still air and drawn. There is no ferritic outline of the austenitic grain but from the markings it is judged that the prior austenitic and the existing grains are the same.

The accompanying Fig. 3 is item 317, the strongest in creep test. The formations and boundaries and more especially the history of the steel given later indicate that the existing grain is a fractional part of the prior austenitic grain.

The accompanying Fig. 4 is item 362. The structure is due to an oil quench and draw so there is no question of the cementite being in a sorbitic form.

These microstructures were polished and etched to show the dispersion of the carbides. This steel usually requires a different degree of polish and etch to show definite grain boundaries.

The chemical analysis and physical properties at room temperature are listed in the accompanying Table I. The Charpy bar has the keyhold notch described in Metals Handbook, 1936 Edition, American Society for Metals, p. 50.

The history of the steels are of interest as items 315, 319, and 317 were made from the same heat of steel but differ in amount of hot forge work. The steel is basic electric, cast in a 21-in. corrugated ingot and forged to a 10-in. square annealed billet. The billet was reforged to a 6-in. octagon, a total forge reduction in area of 12 to 1. Item 319 was cut from a mid-radius of the section as a 1-in. square bar, then normalized and drawn. Another billet length was forged to 1-in. square bars, a forge reduction of 350 to 1. The bars were heat treated for items 315 and 317. As both test items have the same history up to the cooling in the final heat treatment, one would expect the bars to have the same prior austenitic grain shown in the accompanying Fig. 1.

TABLE I.—COMPOSITION AND PHYSICAL PROPERTIES.

Item Number	Chemical Analysis, per cent					Physical Properties at Room Temperature					
	Carbon	Nickel	Chromium	Molybdenum	Manganese	Tensile Strength, lb. per sq. in.	Elastic Limit, lb. per sq. in.	Elongation in 2 in., per cent	Reduction in Area, per cent	Charpy Impact	Test
315....	0.32	1.92	0.75	0.47	0.58	125 300	53 000	18.0	50.3	16.3	Before
						132 700	74 000	20.6	54.5	14.5	After
319....	0.32	1.92	0.75	0.47	0.58	....	....	....	....	....	Before
						122 100	80 000	22.8	54.9	8.0	After
317....	0.32	1.92	0.75	0.47	0.58	120 800	80 000	21.5	60.6	24.9	Before
						128 900	89 000	18.6	60.0	22.8	After
362....	0.37	1.77	0.69	0.33	0.66	168 200	137 000	17.0	60.1	28.8	Before
						168 000	154 000	20.0	59.1	29.9	After

Item 362 is from a basic electric steel cast in a 11½-in. square ingot of 1270 lb. weight and rolled to 4-in. round bars, oil quenched and drawn.

While the history of the steel is of interest to the steel maker, the fabricator must heat treat to the service requirements of the consumer. The final heat treatment is influenced by the condition of the steel and pre-heat treatments are often used to obtain desired grain size results. The details of, more than the methods of, deoxidization are important. There are commercial steels with grain growth – temperature and grain growth - time characteristics which require impracticable heat treatments to obtain the desired structures for higher temperature service.

Dilation curves on a number of steels with the chemical analysis in Table I gave the end of the reaction at 375 C. (710 F.) for cooling rates of 135 C. (240 F.) and 10 C. (18 F.) per hour. The higher rate gave one dilation beginning at 500 C. (930 F.) and the lower rate two dilations beginning at 675 C. (1250 F.) and 470 C. (880 F.).

The upper minimum time on the *S* curve, that is, the smallest total time for an isothermal transformation to produce lamellar pearlite is 15 hr. when held at 675 C. (1250 F.). The corresponding time for an eutectoid carbon steel is less than that number of seconds.

These values indicate the extreme sluggishness of this steel in transformation, the reasons why sorbite was obtained in the "annealed" steel of Fig. 1 and the practical difficulties of obtaining prior austenitic grain sizes.

Tests give the lowest temperature of recrystallization to be near 470 C. (880 F.) but below 480 C. (900 F.). The creep tests considered in the paper were "step down" tests of 2000 to 3000 hr. duration, not extrapolated, and run at 450 C. (840 F.) which is below the recrystallization temperature. The relation of the testing temperature to the lowest temperature of recrystallization can have a profound influence upon the characteristics indicated in this paper.

In the Fig. 2 plot of creep strength against the existing grain size there is agreement that the creep strength increases with the size of the grain. In the discussion it is suggested that the creep strength should continue to increase up to the limit of the steel in producing coarse grains. But the correlation of data in Fig. 2 bends the curve to a maximum, then droops to a decreased value. It is the cause of the bend in the curve that has provoked discussion.

Figure 2 considers only two variables, creep stress and grain diameter. For a smooth curve any variables that enter the problem must be functions of creep stress or grain diameter. Simply stated, the variables in creep can be referred to the slip planes contained in each grain and considered in terms of the area and of the circumference of the planes. The area of the slip planes in a row or layer of grains, projected upon a unit area for the principal tension stress, is of unit area and independent of the size of grains but dependent only upon the strength of the material in the slip plane. The area of each slip plane is bounded by a circumference of the grain and the resistance to slip due to contact between grains is proportional to grain diameter.

The material within the grain and in the slip planes is a function of creep strength only. For the same chemical composition of steel, the strength may vary due to the type and degree of carbide dispersion as suggested by Miller and Aborn and later shown in the accompanying Figs. 1 to 4, or to the submicroscopic forms of carbides of iron and alloy as discussed by McKinney. The author considered all of the structures as sorbitic, without any free or rejected ferrite and not greatly differing in creep strength within the volume of the grain. To project the curve in Fig. 2

without a bend it would pass beyond any tensile strength properties for the larger grained test. We cannot reconcile the differences in carbide distribution or the tensile properties of the steel with the much greater difference between the projected and actual creep strengths. Variables that enter through the area of the slip planes can only account for a small part of the causes producing the bend in the curve.

The grain boundary effects in polycrystalline materials has long been recognized. Various hypotheses are made to suit known properties. The boundary material has been described as an "amorphous" steel with cohesive properties different from the steel within the grain but with equal strengths at the "equicohesive temperature." The grain boundary has also been considered as due to irregularities and discontinuities in the atomic space lattice where one grain lattice system contacts another, and influenced by "self-diffusion" and "lowest temperature of recrystallization." Crystallographic studies of various metals as lead, tin, and zinc are becoming more current in recent scientific literature.

With the influence of the grain boundary or edge of the slip planes as a recognized phenomenon, the strength of a polycrystalline metal would naturally include a function varying with the grain diameter. The strength of metal contained within the grain or the area of the slip planes, measured as a unit stress, would be a function independent of the grain boundary or grain size. With these variables a curve like Fig. 2 would approach a straight line only under special or limiting conditions.

It is a general rule that, at the lower temperature for creep, finer grained steel shows the better creep resistance. If this lower temperature condition is plotted on Fig. 2 the curve would approach and lie along the vertical coordinate axis for stress. It is observed that at the higher temperatures for creep the coarser grained steel has the better creep resistance. If this condition is plotted on Fig. 2 the higher temperature curve should lie near the horizontal coordinate axis for grain diameter. If curves are drawn for different constant temperatures between these limiting conditions, one could not expect the greatest creep strength to be obtained with the finer grained steel for the temperature range between the lowest creep temperature to the "lowest temperature of recrystallization," and then a jump to the coarser grained steel for the range between the lowest temperature of recrystallization to the higher creep temperature. A transition in grain size for the greatest creep strength at each temperature should be found between the limiting conditions of finer grain for lowest temperature and of coarser grain for higher temperature. The transition implies not only a curve similar to Fig. 2 which is near the lowest temperature of recrystallization, but a family of constant temperature curves having a similarity in shape.

Many variables enter into the creep problem so that these deductions represent general characteristics and trends. A number of series of grain size special tests run over the past several years to check these conclusions and to obtain numerical results are in preparation for presentation at an early date.

AN INVESTIGATION OF THE EFFECTS OF ELEVATED TEMPERATURES ON THE FATIGUE PROPERTIES OF TWO ALLOY STEELS

By J. S. Kinney[1]

Introduction

This paper presents partial results of an investigation conducted at the Rensselaer Polytechnic Institute in conjunction with the Research Laboratories of the General Electric Co. and for the Turbine Department of that company to determine the effects of elevated temperatures on the fatigue properties of two alloy steels used in the construction of steam turbines.

The materials tested were: (1) a 0.27 per cent carbon steel containing 1.99 per cent nickel, 0.86 per cent chromium, and 0.41 per cent molybdenum, and (2) a 0.10 per cent carbon steel containing 0.37 per cent nickel and 12.39 per cent chromium.

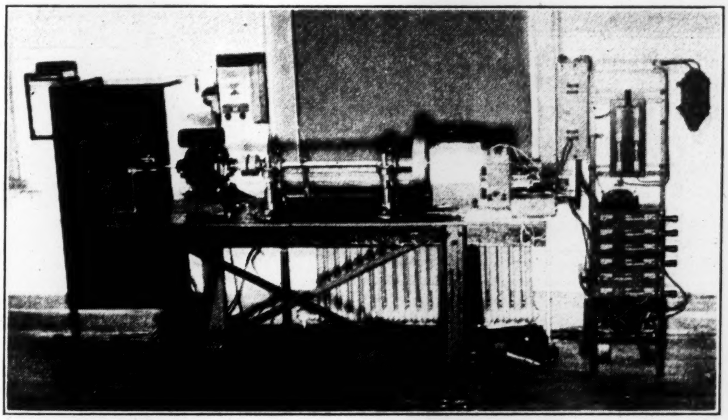

Fig. 1.—Rotating Spring Type Machine.

Equipment

The machine used in conducting these tests was of the rotating spring type, in principle similar to, although somewhat larger than, the machines developed at the University of Illinois.[2] Figure 1 is a general view of the machine and Fig. 2 shows the loading wheel with load-measuring micrometer in position. Referring to Fig. 2, the load was applied to the end of the specimen by a spring, the magnitude being adjusted by means of a large spring adjusting screw set in the rim of the loading wheel. The actual load applied was determined by measuring the deflection of the spring with the micrometer. The desired load having been applied to the specimen, the loading arm was clamped in position with the three clamping bolts. The micrometer, of course, was removed during the running of a test. The loading spring was calibrated by means of dead weights and was checked at regular intervals during the running of the tests. No change was noticed in the various calibrations.

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[1] Formerly at Rensselaer Polytechnic Institute, Troy, N. Y.

[2] H. F. Moore and T. M. Jasper, "An Investigation of the Fatigue of Metals," *Bulletin No. 152*, Engineering Experiment Station, University of Illinois (1925).

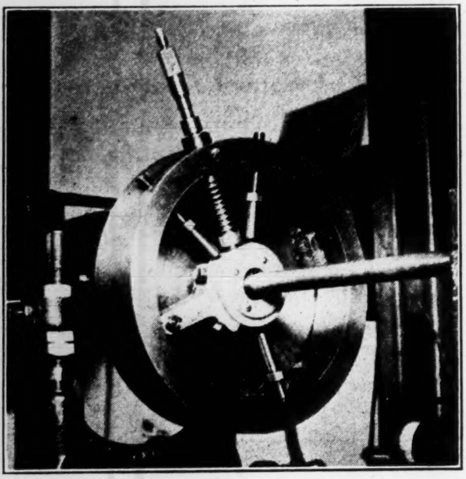


FIG. 2.—Loading Wheel and Load-Measuring Micrometer.

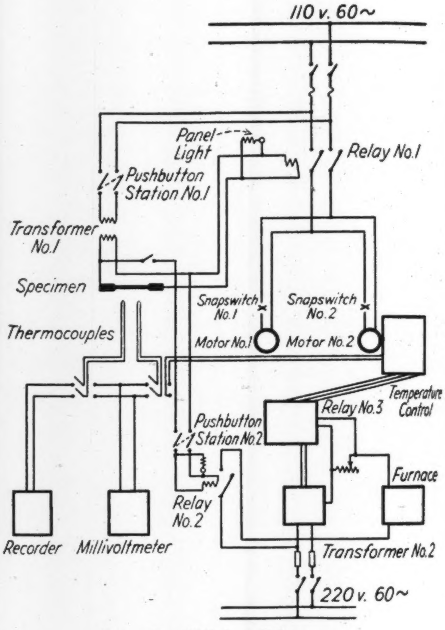
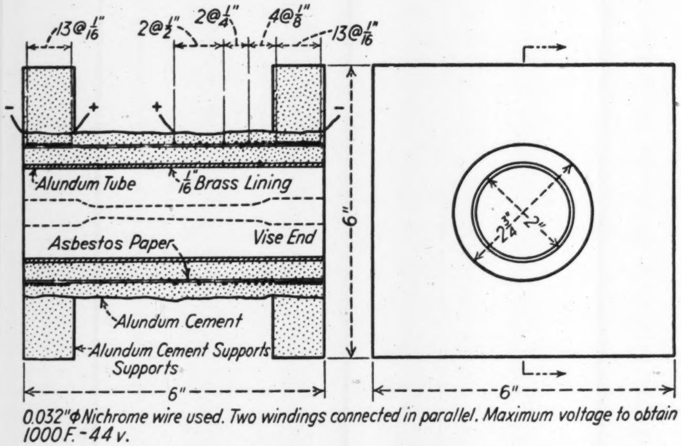

110 v. 60~
Panel Light
Relay No.1
Pushbutton Station No.1
Transformer No.1
Specimen
Snapswitch No.1
Snapswitch No.2
Thermocouples
Motor No.1
Motor No.2
Temperature Control
Relay No.3
Pushbutton Station No.2
Furnace
Relay No.2
Recorder
Millivoltmeter
Transformer No.2
220 v. 60~

FIG. 3.—Wiring Diagram.


13@1/16"
2@1/2"
2@1/4"
4@1/8"
13@1/16"
-
+
+
-
Alundum Tube
1/16" Brass Lining
Asbestos Paper
Vise End
Alundum Cement
Alundum Cement Supports
6"
6"
6"
2 3/4"
2"

0.032"ϕ Nichrome wire used. Two windings connected in parallel. Maximum voltage to obtain 1000 F. - 44 v.

FIG. 4.—Furnace Heating Element.

The machine was driven by a $\frac{3}{4}$-hp. motor at a speed of 1725 r.p.m. This motor is designated on the wiring diagram (Fig. 3) as motor No. 1. Motor No. 2 was a small $\frac{1}{30}$-hp. unit attached to the temperature controller. Power for these motors was supplied through relay No. 1. This relay was operated with power supplied from transformer

No. 1 which delivered current at 24 v. It will be noticed that this circuit contained the specimen itself. Relay No. 2, which controlled the power supply to the furnace, was also operated with power from transformer No. 1. It is thus seen that when the specimen fractured, the power was cut off for both motors and the furnace. The two control thermocouples fastened to all specimens run at high temperatures were connected, one to the temperature controller, and the other to the temperature recorder. The power for the furnace was supplied by means of a variable voltage transformer which made it possible to change voltages from 1 to 45 v. in 1-v. intervals.

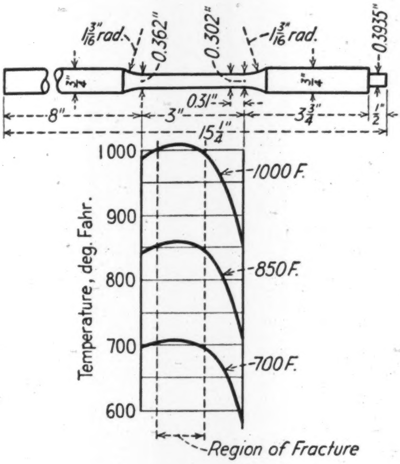


FIG. 5.—Specimen and Temperature Gradients.

The construction of the heating element used in the electric furnace is shown in Fig. 4. The cast alundum cement supports fitted inside a sheet metal container. The space between these supports was filled with diatomaceous earth covered with asbestos board. The sheet metal container provided a space 1 in. long on each end of the heating element which was packed with crude asbestos during the running of a test.

The two control thermocouples previously mentioned were of the chromel-alumel type, and were wired to the specimen side by side with their tips on the tapered section of the specimen and just barely in contact with its surface. Obviously, these couples always had to be placed in exactly the same position on every specimen. The specimens themselves were always placed in the correct longitudinal position relative to the applied load by locating them with the tip of the bearing end just flush with the inside edge of the inner race of the bearing.

CONDITIONS OF TESTS

Endurance limits were determined for the two alloy steels at room temperature, 700 F., 850 F., and 1000 F. All tests were made at a speed of 1725 r.p.m.

SPECIMENS

The specimens used were as indicated in Fig. 5. They were finished by polishing circumferentially with No. 000 emery. This figure also indicates the temperature gradients for the tapered section of the specimens at the three elevated temperatures used. All specimens fractured within the region included between the two dotted lines on Fig. 5. The specimens were of two alloy steels:

Nickel-Chromium-Molybdenum Steel.—The chemical composition and physical properties of this steel were as follows:

COMPOSITION, PER CENT	
Carbon	0.27
Phosphorus	0.015
Sulfur	0.011
Silicon	0.25
Nickel	1.99
Chromium	0.86
Molybdenum	0.41

PHYSICAL PROPERTIES	
Tensile strength, lb. per sq. in.	130 200
Yield strength, lb. per sq. in.	86 000
Elongation in 2 in., per cent	23.2
Reduction of area, per cent	67.2

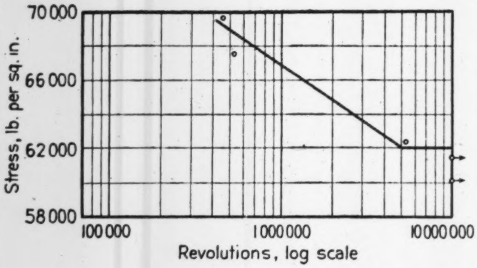


FIG. 6.—Nickel-Chromium-Molybdenum Steel, Room Temperature.

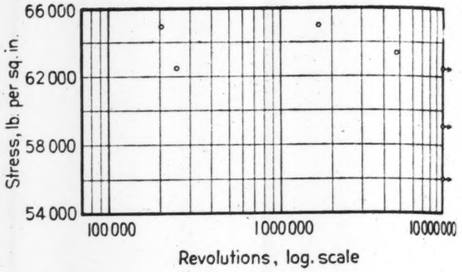


FIG. 7.—Nickel-Chromium-Molybdenum Steel, 700 F.

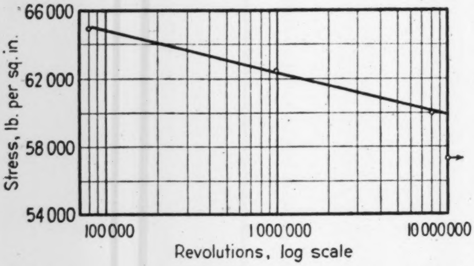


FIG. 8.—Nickel-Chromium-Molybdenum Steel, 850 F.

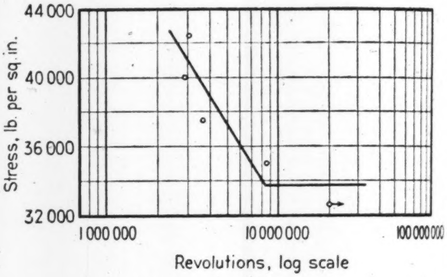


FIG. 9.—Nickel-Chromium-Molybdenum Steel, 1000 F.

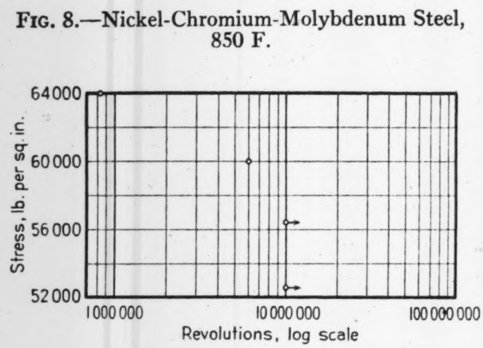


FIG. 10.—12 per cent Chromium Steel, Room Temperature.

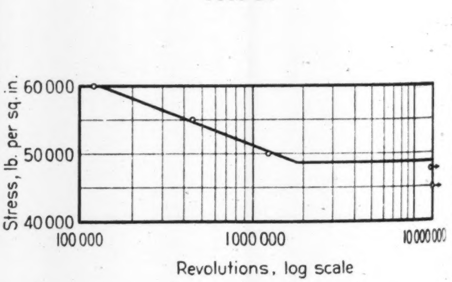


FIG. 11.—12 per cent Chromium Steel, 700 F.

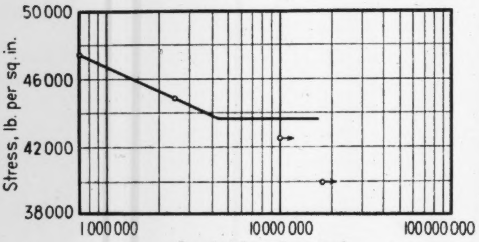


FIG. 12.—12 per cent Chromium Steel, 850 F.

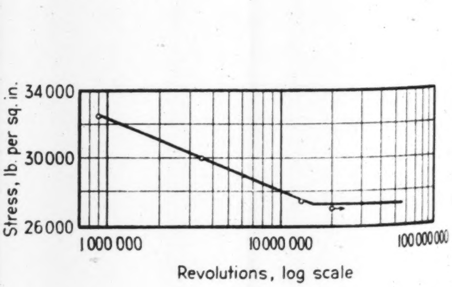


FIG. 13.—12 per cent Chromium Steel, 1000 F.

This material was air cooled from rolling temperature, then heated and held at 1740 F. for 8 hr., oil quenched to room temperature and reheated to 1200 F., and air cooled.

12 per cent Chromium Steel.—The chemical composition and physical properties of this steel were as follows:

COMPOSITION, PER CENT	
Carbon	0.10
Manganese	0.29
Phosphorus	0.014
Sulfur	0.019
Silicon	0.31
Nickel	0.38
Chromium	12.39

PHYSICAL PROPERTIES	
Tensile strength, lb. per sq. in.	111 700
Yield strength, lb. per sq. in.	80 000
Elongation in 2 in., per cent	23.5
Reduction of area, per cent	67.9

This material was air cooled from rolling temperature, then heated and held at 1175 F. for 5 hr., furnace cooled to 570 F., and then air cooled to room temperature.

TEST RESULTS

The *S-N* curves for the various series are shown in Figs. 6 to 13. Obviously in Figs. 8, 12, and 13 the unbroken specimens could have been run to a greater number of revolutions to good advantage. However, with the machine as it existed in this investigation, it was impossible to run one specimen for more than 20,000,000 revolutions, because of the failure of the loading bearing. This failure was caused by the small arcing of the relay operating current as it passed through the bearing. This difficulty can be eliminated.

Endurance limits are shown plotted as a function of temperature in Fig. 14. For comparison, a similar curve is shown in this same figure for an S. A. E. No. 2340 steel. This curve was plotted from results reported by Moore and Jasper.[2] This material had a tensile strength of 164,900 lb. per sq. in., proportional elastic limit of 136,300 lb. per sq. in., elongation in 2 in. of 10.8 per cent, and reduction of area of 58.2 per cent. This steel was heated to 1525 F., held 30 min., cooled in furnace, reheated to 1450 F., held 15 min., quenched in oil, reheated to 800 F., held 30 min. and cooled in air.

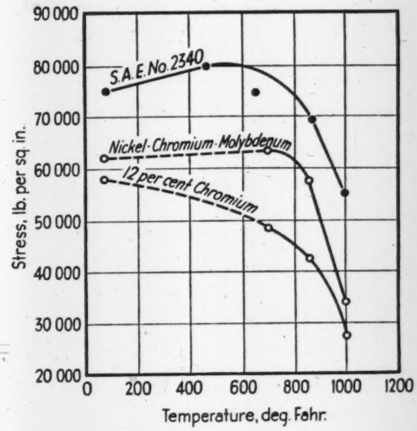


FIG. 14.—Endurance Limit Curves.

NOTE ON THE HARDNESS MEASUREMENTS OF VERY HARD STEELS AND WHITE CAST IRONS

By J. S. Vanick[1] and J. T. Eash[1]

Synopsis

In the use of hardness specifications for the manufacture and purchase of very hard cast irons and steels, difficulties have been encountered in expecting consistent relationships between the several forms of hardness testing employed. In some cases the correlation between hardness values obtained upon hardened steels has been used indiscriminately upon the very hard cast irons. The resulting difficulties led to an exploration of the hardness values obtained upon very hard test blocks of chilled alloy cast irons and upon correspondingly hard steels, all tested in accordance with the standard procedures for hardness testing. Brinell, scleroscope, Rockwell and Vickers tests were made. A result typical of the work shows that the actual Brinell values upon the chilled alloyed cast irons are 40 units harder than those obtained upon steels of the same scleroscope hardness. This difference is reduced somewhat in a comparison of the Brinell with the other indentation hardness tests, such as the Rockwell and Vickers. Reference is made to the further distortion in scleroscope hardness values when the German and American instruments are compared in the range of high hardness values. The results assist in accounting for the advantage in performance which the hardened cast irons have shown in certain applications. The discord in relationships emphasizes the importance of specifying a type of test which possesses a narrower dispersion from the average in individual tests, such as the Brinell, or specifying a sufficient number of individual tests with other methods, with a tolerance on either side of the average sufficient to encompass the expected spread in values.

Regardless of resulting inaccuracies, it has become a growing custom to compare the hardness of various metals by converting Vickers, Shore, Rockwell or Brinell hardness measurements from one scale to another. For most of the ordinary steels, such conversion is fairly satisfactory. Practically all conversion charts have at some time or other been referred to Brinell hardness, probably because the Brinell test is the oldest established means for determining the hardness of metals. Cast iron has frequently been compared across the scales just like steels. In these instances, inherent differences and the elastic properties of cast iron remain uncorrected factors, thus adding to the error already existing in all such conversions.

For most industrial uses, the specification of a Brinell, Rockwell, Shore, or Vickers hardness, along with the composition and one or more additional physical properties has operated fairly enough in procuring material of proper quality. The automotive, machine tool, mining, and metal working industries have been able to specify the composition and a Brinell or Rockwell hardness, and in some cases a Shore hardness, to define their requirements in cast iron. Particularly in the purchase and sale of rolls for the metal, rubber, and paper trades, it has become a custom to specify scleroscope hardness values, and to compare these values interchangeably in terms of Brinell or other hardness values.

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[1] Metallurgists, Development and Research Division, The International Nickel Co., Inc., Bayonne, N. J.

In many of these cases, the structure and composition of the casting have not been disclosed and comparisons between steel and chilled iron rolls, both possessing equal hardness values, have led to considerable confusion. Usually the chilled iron roll excels in performance the steel roll of corresponding hardness. On the other hand, it appears to be almost impossible to develop a scleroscope hardness of 100 in chilled iron rolls, an achievement quite feasible in steel forgings. Furthermore, foreign roll makers, especially those of Continental Europe and particularly Germany, can confuse American roll makers and users with the presentation of higher scleroscope values than are obtained in the United States. This creates a problem that should perhaps be investigated by the International Association for Testing Materials, because in this particular case the instruments employed to obtain the comparison differ by over ten units, enough to cause considerable trouble. If, for example, advice about a chilled roll with a German scleroscope hardness of 100 is interpreted in terms of a 100 U.S. scleroscope hardness value, the product cannot practically be made to this value. Attempts to convert the 100 scleroscope to an equivalent Brinell value from the steel conversion charts lead to grotesque Brinell values of 750 to 800. If, at this time, the German producer is asked to make a roll with a Brinell hardness of 750 the comedy of errors is completed. For these reasons, work has begun at the Bayonne Laboratory of The International Nickel Co., and independently in France[2], to investigate the problem of measuring by various means the hardness of steels and irons which possess high hardness and in comparing the values so obtained.

The usual standard testing procedure was followed for each test. For Brinell values a 10-mm. tungsten carbide ball held at a 3000-kg. load for 30 sec. was used. Two or three determinations proved sufficient to establish closely uniform values. For Rockwell values, the "C" scale and 150-kg. load was used. For Vickers values, the diamond pyramid with a 50-kg. load was used. Rockwell and Vickers tests required an average of 9 and 8 readings, respectively, to narrow the scatter in values. For the scleroscope hardness test, a Shore direct-reading instrument was used, standardized against a steel block calibrated to a value of 95. An average of 16 tests had to be made with this instrument to narrow the scatter in the results.

The difficulties encountered in this investigation are most apparent in the very hard grades of chilled iron, and for that reason nickel-chromium or nickel-chromium-molybdenum alloyed chilled irons of the Ni-Hard type were selected to cover a range of 500 to 750 Brinell, 550 to 950 Vickers, 45 to 65 Rockwell "C" and 60 to 95 scleroscope. Tests were made upon spalls from the surfaces of five commercial nickel-chromium type rolls, and upon twenty chill-cast iron test bars of varying nickel-chromium

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[2] M. Ballay and R. Chavy, "Les Fontes Blanches Speciales Researches sur les Proprietes de Quelques Fontes Blanches au Nickel-Chrome," *Bulletin* de l'Association Technique de Fonderie, Vol. 9, November, 1935, pp. 366-380. French paper presented at the International Foundry Congress, Brussels, September, 1935.

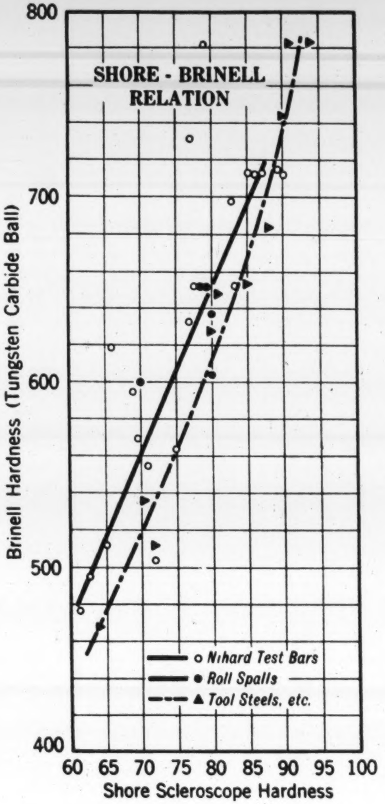

Fig. 1.—Brinell - Shore Scleroscope Hardness Relation.

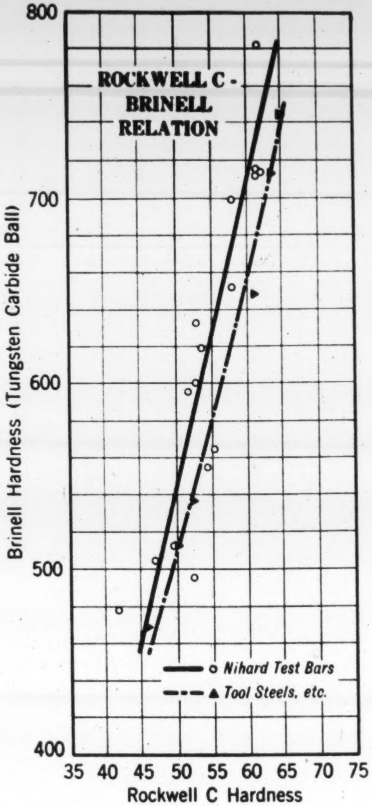

Fig. 2.—Brinell - Rockwell, "C" Scale, Hardness Relation.

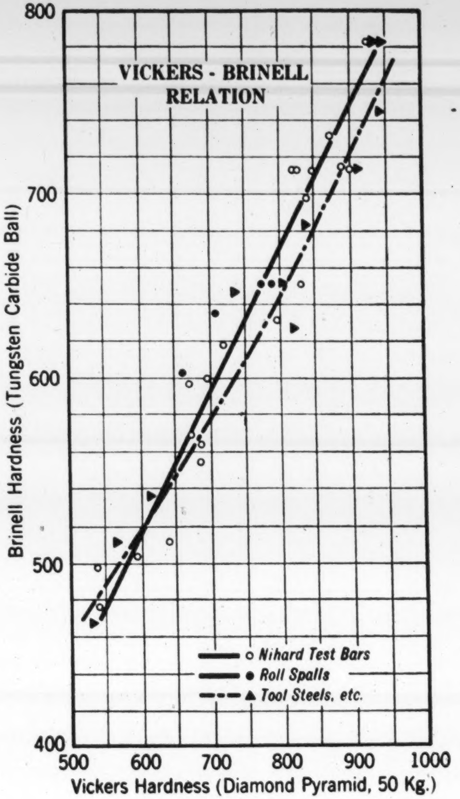

Fig. 3.—Brinell - Vickers Hardness Relation.

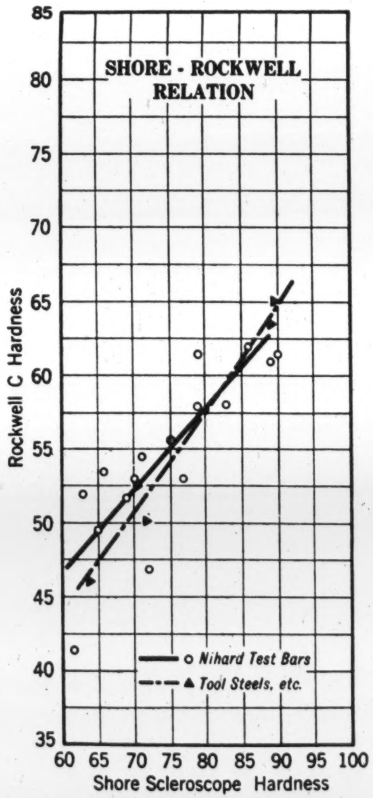

FIG. 4.—Rockwell, "C" Scale - Shore Scleroscope Hardness Relation.

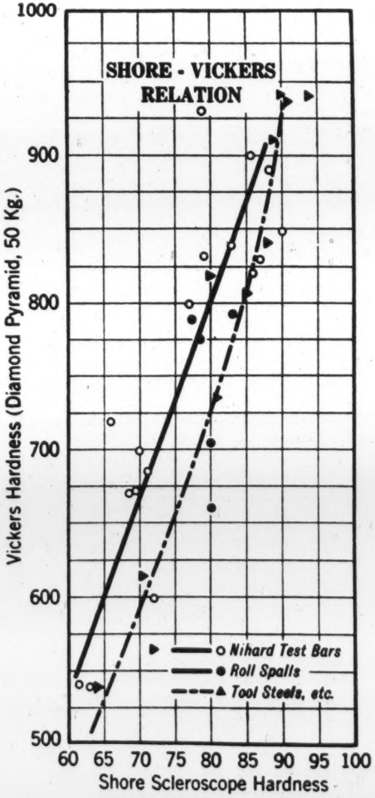

FIG. 5.—Vickers - Shore Scleroscope Hardness Relation.

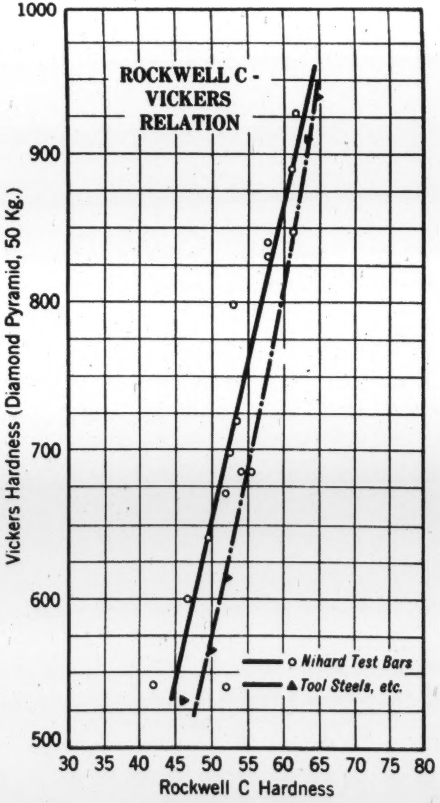

FIG. 6.—Vickers - Rockwell, "C" Scale Hardness Relation.

TABLE I.—ANALYSES AND HARDNESS OF CHILLED IRONS AND HEAT-TREATED STEELS.

Material Tested	Total Carbon, per cent	Silicon, per cent	Nickel, per cent	Chromium, per cent	Manganese, per cent	Shore Hardness Readings, Average Values	Vickers Hardness Readings, 50-kg. Diamond Crystal, Average Values	Brinell Hardness Readings, 3000-kg. Load,[c] Average Values	Rockwell Hardness, "C" Scale, Readings, Average Values
NICKEL-CHROMIUM CHILLED IRON ROLL SPALLS:									
Roll spall No. 1	....	....	....	....	....	78.5	774	652	....
Roll spall No. 2	....	....	....	....	....	83.3	792	652	....
Roll spall No. 3	....	....	....	....	....	77.5	788	652	....
Roll spall No. 4	....	....	....	....	....	80	660	604	....
Roll spall No. 5	....	....	....	....	....	80	770	637	....
NICKEL-CHROMIUM CHILL CASTINGS:									
Low alloy nickel-chromium chill block	....	....	....	....	....	69	672	570	....
Chill block	3.64	0.60[a]	5.30	1.63	....	87	829	713	....
	3.62	0.60[a]	5.27	1.53	....	86	820	713	....
	....	....	....	....	....	77	874	732	....
Bar (1¼-in. diameter)	3.21	0.52	5.96	1.12	0.30	61.5	542	477	41.7
	3.47	0.87	4.54	1.73	0.36	79	929	782	61.7
	3.30	0.13	3.55	1.73	0.32	65.1	642	512	49.6
Chill block	3.64	0.60[a]	5.30	1.63	....	89.3	889	715	61.1
	3.62	0.60[a]	4.50[a]	1.50[a]	....	90	848	713	61.5
	3.65[a]	0.60[a]	5.34	1.87	....	85.9	900	713	62
Bar (1¼-in. diameter)	3.41	1.01	6.53	1.64	1.43	72	597	504	46.8
	3.36	0.13	6.54	1.59	0.29	83	838	698	58.0
	3.36	0.13	6.54	1.59	0.29	79	831	652	58.0
	3.36	0.13	6.54	1.59	0.29	71	685	555	54.4
	3.21	0.52	5.96	1.12	0.30	70	698	600	52.8
	3.30	0.13	3.55	1.73	0.32	77	797	632	53.0
	3.25	0.19	5.58	1.58	0.30	75	686	564	55.5
	3.41	1.01	6.53	1.64	1.43	63	539	495	52.0
	3.50	1.04	3.57	1.74	1.50	69	671	595	51.8
	3.50	1.04	3.57	1.74	1.50	66	719	619	53.5
HEAT-TREATED STEEL:									
No. 0511 - tool steel - water quenched, 1450 F.	0.97	0.20	....	....	0.27	94	940	782	....
	....	....	....	....	....	91	935	782	....
No. 0518 - tool steel - water quenched, 1600 F.	1.08	0.20	0.17	0.49	0.16[b]	80	818	627	....
	....	....	....	....	....	85	806	652	....
No. 0512 - tool steel - water quenched, 1550 F.	1.10	0.20	....	....	0.28	88	840	683	....
Tool bit	....	....	....	....	....	90	940	744	65
	....	....	....	....	....	89	910	713	63.5
Oil-quenched steel	....	....	....	....	....	64	531	468	46
	....	....	....	....	....	81	736	647	61
	....	....	....	....	....	72	565	512	50
	....	....	....	....	....	70.5	614	536	52.5

[a] Approximate.
[b] 1.60 per cent tungsten.
[c] Tungsten carbide ball.

content. Eleven very hard tool steels, taken from laboratory stock as well as from commercial production, were quenched and tempered to produce a high hardness within the top ranges. The results of these tests appear in Table I and in Figs. 1 to 6. Average values were plotted. It should be emphasized that the object of the work was to report the results obtained upon these very hard materials when tested by using standard hardness testing methods rather than to investigate the shortcomings of hardness of testing machines, or the inhomogeneity in material which the scatter in results clearly indicated.

DISCUSSION OF RESULTS

The most significant feature of the charts is that the solid line representing the Brinell hardness values of the chilled cast iron is in all cases above the line representing the hardness of the steel. This conclusion is made notwithstanding the fact that average values on an individual casting may depart by a sizeable margin from the line representing the average hardness for the entire series of irons, as illustrated in Figs. 1, 2, and 3.

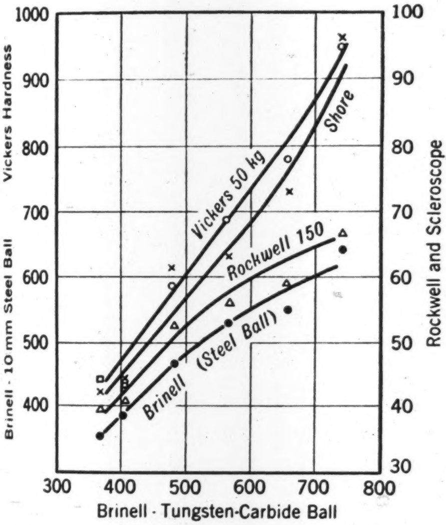

FIG. 7.—Comparison of Hardness Values of Chilled White Iron as Determined by Ballay and Chavy.

In the higher hardness range, the chilled iron seems to depart further from the average line towards abnormally high Brinell values as illustrated in Fig. 1. The possibility of appreciable amounts of austenite in the chilled iron may account for a lowered degree of elasticity and consequently low scleroscope values. A further indication of the difficulty in obtaining reproducible values, particularly in the case of the chilled cast iron, is shown by the number of readings needed to narrow the spread in values. These indicate that the Brinell is least affected, the Rockwell and Vickers next, and the scleroscope most.

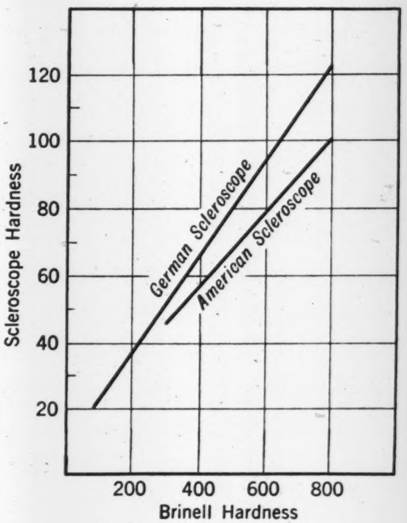

FIG. 8.—Relation Between German and American Scleroscope Values.

The values plotted in Fig. 3 (Brinell and Vickers) and the values shown in Fig. 4 (scleroscope and Rockwell) are bracketed within a narrow enough band to eliminate any significant differences between nickel-chromium chilled iron and the forged steels. To all practical purposes this would mean that a conversion within these two charts may be performed by using essentially the same conversion factor for both the nickel-chromium chilled iron and the steel. The dispersion of scleroscope values in the case of Fig. 4 is so broad that it appears to be the least reliable form of test. The relations between Vickers, Rockwell and Brinell data, as expressed in Figs. 2, 3 and 6, seem to be more consistent, undoubtedly due to similarity in action of the indentation type of test.

The conversion of the scleroscope to the indentation type of test, as illustrated in Figs. 1 and 5, differs in the case of the two materials. Scleroscope values obtained on the forged steel are 5 to 7 points higher, as shown in Figs. 1 and 5, than those obtained on nickel-chromium chilled iron of the same hardness. This difference is important and must be taken into account when comparing the scleroscope hardness of forged steel rolls with chilled iron rolls. For equivalent scleroscope hardnesses, the nickel-chromium chilled iron rolls will have a Brinell hardness exceeding that of the forged steel by about 40 units. Conversely for equivalent Brinell hardnesses, the roll will record a Shore value 5 to 7 points lower than that of forged steel.

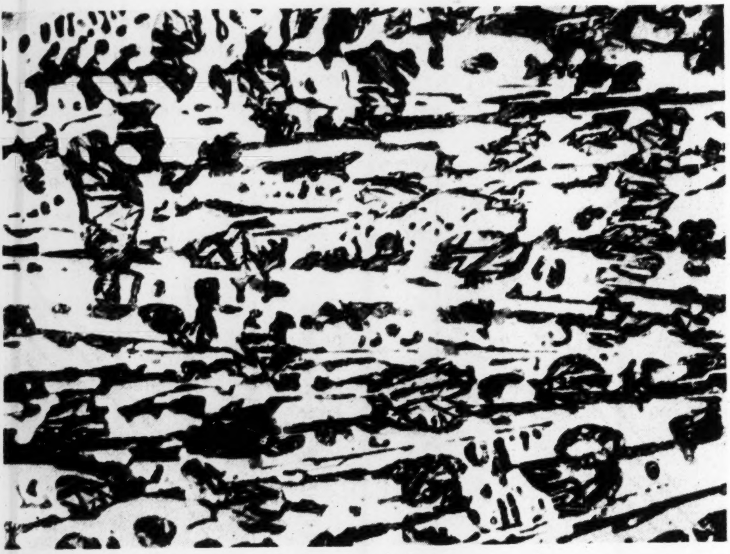

FIG. 9—Distribution of Carbide and Martensite in Chilled White Cast Iron of the Nickel-Chromium Type (× 500).

Etched in 2 per cent Nital.

Total carbon, per cent	3.53
Silicon, per cent	1.01
Manganese, per cent	0.43
Sulfur, per cent	0.08
Phosphorus, per cent	0.17
Chromium, per cent	1.50
Nickel, per cent	4.66

If the area represented above were compared to the area of the Brinell impression in a relative way, it would occupy about the size of a postage stamp in a wash basin 12 in. in diameter. Actually, if the indentations made by the different instruments on a chilled cast iron having a Brinell hardness of 650 were magnified to the same extent of the photomicrograph (× 500) they would have the following diameters:

Brinell	650	48 in. diameter
Rockwell, "C" scale	58	8.75 in. diameter
Vickers, 50-kg. load	775	6.8 in. square
Scleroscope	80	6.25 in. diameter

The work done by Ballay and Chavy[2] upon this problem is illustrated in Fig. 7, where their comparison of the hardness values obtained upon chilled white iron is illustrated. This diagram does not compare cast iron with steel, but does show that at the upper hardness levels the Rockwell and Brinell tests tend to depart from their straight-line relationship at lower values. A comparison between hardness values shows among other things the relation between the steel ball and tungsten carbide ball in the Brinell test upon these very hard irons. In Fig. 8, the work of Schuz[3] compares the German and American scleroscope tests upon hard materials, and in the range around 80 on the American scleroscope the German instrument is shown measuring 10 to 15 units higher values for the same Brinell hardness.

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[3] E. Schuz, "Über die Harte und Schrecktiefe des Legierten Schalenhartgusses," *Die Giesserei*, Heft 31-32, pp. 321-327 (1934).

As stated above, it is not the purpose of this paper to enter into an extensive discussion of the irregularities in the materials or in the instruments. Chilled cast iron is admittedly an inhomogeneous material and is, therefore, expected to vary in the tests, depending upon the microstructure and the area that is integrated into the final result. Thus the structure, as shown in Fig. 9, is composed of at least two principal components. It is difficult to concede the comment frequently made that the scleroscope point strikes only one single component. The relative areas covered by the hardness instrument in making a test corresponding to a value of 650 Brinell, 58 Rockwell, 80 Shore and 775 Vickers is illustrated in Fig. 9. It is obvious that the Brinell (tungsten carbide) impression embracing the greatest area is likely to yield the most consistent values.

In view of the fact that the scleroscope is widely used to measure the hardness of heavy and cumbersome pieces, such as rolls, the utility of this instrument must be recognized. Again the inhomogeneity of the cast iron indicates that this instrument, depending upon a very small area for its record, may chance upon spots which differ widely in mineral hardness as well as in elastic and strength properties, so that the final value selected must encompass a large enough number of tests to balance these factors and arrive at a statistical average. The importance of this feature is readily recognized in the case of a nickel-chromium chilled cast iron which is carefully controlled to obtain a martensitic body enmeshed in an iron-carbon-chromium framework, but the hardness value obtained may be affected depending upon whether the metal tested is rich in carbide or in martensite. A variation in quantity of carbide may occur due to the 2.5 to 3.5 per cent range of carbon contents utilized in chilled iron roll manufacture. The steels are less subject to this difficulty, partly because the carbide component is more thoroughly dispersed and is present in small insignificant quantities. Furthermore, the effect of forging tends to make the structure more dense and close out microscopic porosity which might affect a hardness test.

CONCLUSIONS

Results of these tests show that the dependence upon scleroscope hardness for a measure of hard steel or hard white iron castings is subject to a considerable risk of error due to the highly selective character of the test. Brinell hardness values obtained by conversion from the scleroscope results on cast iron are not always duplicated in actual Brinell tests. This work has shown that the Brinell hardness measured on chilled rolls of the nickel chromium cast iron type will exceed the Brinell hardness obtained upon forged steels of the same scleroscope hardness by approximately 40 Brinell units. Conversely, at the same Brinell hardnesses, the chilled hard iron will possess a scleroscope value 5 to 7 points lower than that of a forged hardened steel.

In the range of 65 to 85 scleroscope, the nickel chromium chilled iron Vickers hardness values are approximately 65 units higher than tool steel of the same scleroscope hardness. The scleroscope to Rockwell and Vickers to Brinell comparisons seem to maintain a fairly consistent relationship.

The hardness of rolls is often used as an indication of their expected performance, and in general the performance in service of rolls of the same general type increases with an increase in hardness. The Shore scleroscope should not be used, however, as in indication of performance when comparing chilled iron rolls with forged steels rolls for observations have been made in many practical applications where the output of a hard chilled iron having a scleroscope hardness of 90 excels the output of a forged steel roll with a scleroscope hardness exceeding 100. It is evident, therefore, that the scleroscope values are not a reliable index of the performance nor of the comparative hardness in the very high hardness ranges.

German scleroscope values are not directly equivalent to results obtained with American instruments but are 10 to 15 units higher in the high hardness range. This conflict in scleroscope hardness between German instruments and those of other countries should be composed through the International Association for Testing Materials to avoid the confusion inevitably resulting from direct translation of German values without adequate definition or the use of a suitable conversion factor.

Acknowledgment.—The authors wish to acknowledge the effort and work done upon this problem by T. J. Wood, now associated with the Robins Conveying Belt Co., Passaic, N. J.

DISCUSSION

Mr. L. B. Tuckerman.[1]—I have nothing but praise for the very careful work, the very careful comparisons and the useful results which have come out of this thorough study of the relation between the readings of various instruments supposed to measure a something called hardness. The only criticism I would make relates to the implication in some of the words of the authors. In the paper I noticed this statement: "The scleroscope values are not a reliable index of the performance nor of the comparative hardness in the very high hardness ranges." I said to myself "Can it be that they are still thinking that there is such a thing as absolute hardness? No! That must be just a case of careless wording." But in presenting the paper Mr. Vanick referred to "absolute measurement of the hardness properties."

A good many years ago when I was a professor of theoretical physics I had heard of something known as hardness and naively thought it would be possible to devise a method by which the hardness of materials could be measured. The word sounded good, and it was a property of materials which people wanted to know. So, when I came to the National Bureau of Standards, I thought I would find out how to measure hardness, but immediately found myself in all sorts of difficulties.

Finally, some thirteen years ago, after having worried about it and studied the work of Hertz, Auerbach and Brinell and many others, and made many tests in the laboratory, I wrote a squib on "Hardness and Hardness Testing."[2] In it I made certain statements that I wish to repeat here: "'Hardness' in common parlance represents a hazily conceived conglomeration or aggregate of properties of a material more or less related to each other. These properties include such varied things as resistance to abrasion, resistance to scratching, resistance to cutting, ability to cut other materials, resistance to plastic deformation, high modulus of elasticity, high yield point, high strength, absence of elastic damping, brittleness, lack of ductility and malleability, high melting temperature, magnetic retentivity, etc. This confusion under the one designation 'hardness' results from the fact that there is a rough parallelism in these properties in a large number of materials. The fact that 'hardness' thus conceived, is a conglomeration of different, more or less unrelated properties makes it impossible to correlate any one definite, measurable property with all the current implications of hardness.

"This does not mean that under the hazy conglomeration of properties which are included in the common understanding of hardness, there are not included very important properties of the material."

People come to us at the National Bureau of Standards and say "Give us a conversion table by which we may convert Rockwell hardness into Brinell hardness, scleroscope hardness, etc.," and we say "Gentlemen, we don't know how to do it." And they say "Are you fooling? You know how to convert centimeters into inches, perhaps you would know how to convert inches into miles; why can't you tell us how to convert scleroscope hardness into Brinell hardness?" And we say "That is something different."

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[1] Assistant Chief, Division of Mechanics and Sound, National Bureau of Standards, Washington, D. C.

[2] *Mechanical Engineering*, Vol. 47, No. 1, January, 1925, pp. 53–55.

In algebra there is a property of numbers which we like. We like to think: "If I have a number A and the number A is bigger than a number B and the number B is bigger than a number C, then it follows that A is bigger than C." For specimens of a uniform type of material, such as a carbon steel, which differ only in the heat treatments to which they have been subjected, it is possible to find a relation converting various "hardness" values into other "hardness" values which has this comfortable property. But no such relation exists for such different materials as cast iron and steel. Any number of pairs of pieces of materials, say A and B, can be found for which a 10-mm. ball under a 300-kg. load will give a larger indentation on B than on A, but a 3000-kg. load on the same ball on the same two pieces will give a larger indentation on A than on B, so that A is harder than B and B is harder than A. That is very charming and I do not know that anybody has any particular objection to it, but we cannot say that both measurements measure the same property of the materials.

I do not believe that Mr. Vanick or Mr. Eash would care whether the "hardness" of the rolls were 200, 800, or even 10,000 on the Brinell scale, were it not for the fact that "in general the performance in service of the rolls *of the same general type* [italics mine] increases with an increase in hardness." Because of that fact, their study of "hardness" tests has been a useful aid in controlling the manufacture of the rolls. It is evidently very useful and will aid them in the future to manufacture even better and more durable rolls. Their paper would be altogether admirable were it not for the unfortunate implication about "absolute hardness."

At the National Bureau of Standards we have learned to be wary in the use of the word hardness. When we are careless and use the word thoughtlessly we frequently find ourselves in trouble, become entangled in misunderstandings and loose thinking. Some of us have finally decided that the best way out of the difficulty is to follow a suggestion made by C. E. Davies[3]: "Might it not be advisable .... to drop entirely the use of this term [hardness] and to speak of Brinell numbers, Herbert numbers, etc., rather than of Brinell hardness?"

Brinell, Vickers, Rockwell, and other indentation tests on materials, properly correlated with manufacturing and service records rank among the most useful tests which we make on metals. The tests will be none the less useful if we call the results "numbers." If you want to report the result of a Brinell test as Brinell hardness, all right, do so, but do not think that by calling it so you are making it the true absolute hardness of the material. There is no such thing.

MR. V. E. LYSAGHT.[4]—In connection with this very interesting paper, I should like to call attention to Fig. 6 which shows the relation between Rockwell "C" scale numbers and Vickers numbers and I would like to point out that I find a difference in the reported values as compared with tests made in the laboratories of the Wilson Mechanical Instrument Co. on a large number of hard steels.

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[3] C. E. Davies, "More About Hardness," *Mechanical Engineering*, Vol. 46, No. 8, August, 1924, p. 502.

[4] Engineer, Wilson Mechanical Instrument Co., Inc., New York City.

The relation from the data obtained there is shown in the accompanying Fig. 1.

Although the purpose of the paper was not to present a general conversion curve, nevertheless there is a possibility of the relation being used for conversion and I believe the values for tool steel as shown should be carefully checked.

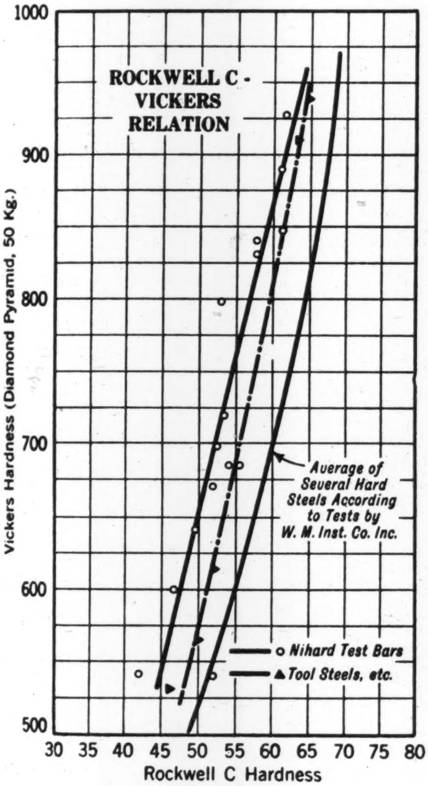

FIG. 1.—Vickers-Rockwell "C" Scale, Hardness Relation.

MR. J. T. MACKENZIE.[5]—I can understand some of the differences in the results reported, but I cannot quite understand why the Vickers, which is a diamond pyramid and the Rockwell which is a diamond cone, should be so different. Also I would like to ask how the modulus of elasticity of the tungsten carbide ball compares with a diamond. Has anybody ever tried a diamond ball, and what is the relative modulus of elasticity of diamond and tungsten carbide?

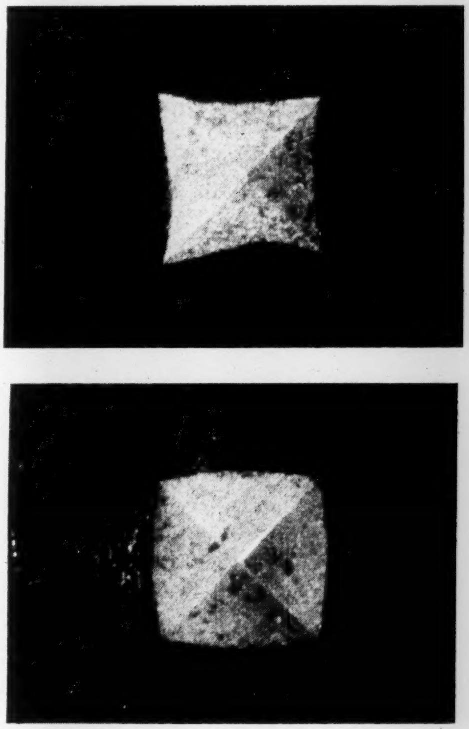

FIG 2.—Vickers Indentations in Copper.
Top—dead "soft."
Bottom—"hard" worked.

MR. TUCKERMAN.—May I take one more moment on this? I wish to emphasize differences in the behavior of different materials in indentation tests which are familiar to all persons who make these tests. The accompanying Fig. 2 shows Vickers indentations made on two pieces of copper, the upper one "dead soft" annealed and the lower "hard" worked. Do you suppose that these two indentations made by the same square diamond prism represent the same or comparable properties of the material?

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[5] Chief Chemist, American Cast Iron Pipe Co., Birmingham, Ala.

Figure 3 shows Brinell indentations made on a cubic face of a copper and an octahedral face of a zinc crystal. Do you suppose that these two indentations made by the same steel ball represent the same or comparable properties of the materials?

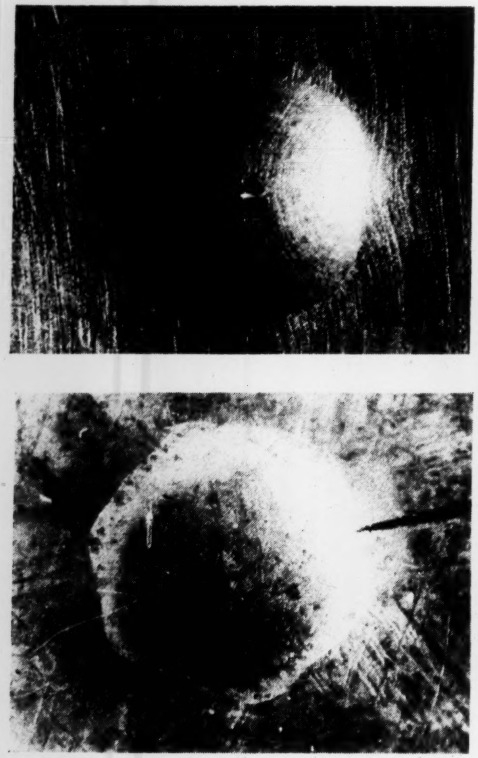

FIG. 3.—Brinell Indentations on Single Crystals.
Top—on a cubic face of a copper crystal.
Bottom—on an octahedral face of a zinc crystal.

Figure 4 shows cross-sections of two Brinell indentations, the upper one on a "dead soft" annealed copper, the lower on a heat-treated aluminum alloy.

Do you suppose that these two indentations made by the same steel ball represent the same or comparable properties of the material? No. They are different and at most only roughly comparable. Illustrations such as these could be multiplied indefinitely.

MESSRS. H. E. FLANDERS[6] AND R. H. HEYER[6] (*by letter*).—Our experiences in testing alloy white cast irons have been quite similar to those of the authors. The difficulties encountered in testing these very hard materials are well known to those working in this field, and the data presented by Vanick and Eash are a valuable addition to the limited information available on hardness tests on this class of material.

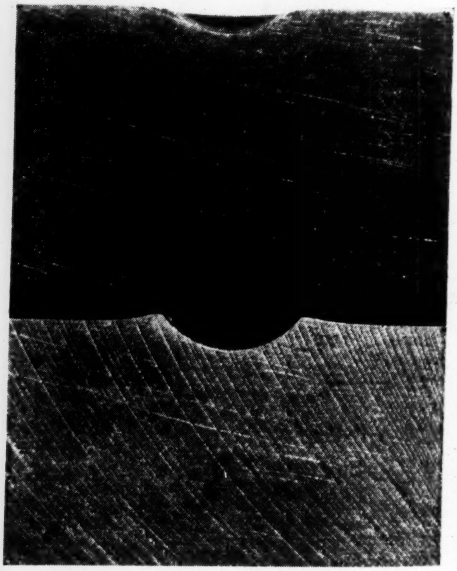

FIG. 4.—Sections of Brinell Indentations.
Top—"dead soft" annealed copper.
Bottom—aluminum alloy.

Certain cast irons having exceptionally high resistance to abrasion have hardness numbers over 66 Rockwell "C" scale despite the fact that numerous radial cracks form about the impression. Cracking in the hardness test can be reduced or eliminated by using smaller loads on the penetrator. Among the tests we have used for these materials are Rockwell "A" and Vickers-Brinell, using 10, 30, or 50-kg. loads. In the latter tests the variations in hardness with test load are of the same magnitude as normal variations for a small sample tested under a given load.

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[6] Supervising Research Engineer, and Junior Metallurgist, respectively, The American Rolling Mill Co., Middletown, Ohio.

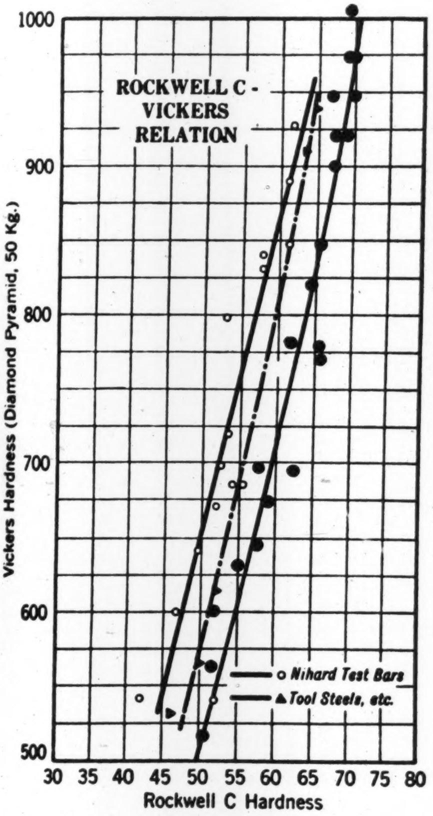

FIG. 5.—Vickers-Rockwell "C" Scale, Hardness Relation.

Comparative tests were made on 25 alloy irons in the cast or cast-and-heat-treated condition using the Rockwell "C" and 10-kg. Vickers-Brinell scales. The results obtained have been plotted on the authors' Fig. 6 as shown in the accompanying Fig. 5. The deviation between the two curves for white irons, averaging about seven points Rockwell "C", indicates that this relationship requires further study before use in converting from one test to the other.

MESSRS. J. S. VANICK[7] and J. T. EASH[7] (*authors' closure by letter*).—Mr. Tuckerman's comments are intensely stimulating, particularly in metallurgical circles that are drifting toward the error of basing all of the properties of a metal upon the results of a "hardness" test. In the light of his discussion, we would agree that all we have to report are the Brinell, Vickers, Rockwell and Shore numbers and concede that no single relation between them exists for different metals. In fact, the wide scatter in the values we obtained suggests that the results should be blocked into bands rather than lines. We presented such results as we obtained hopeful that makers and users of hard castings would allow an adequate tolerance within their specifications to accommodate the unpredictable drift of test results.

This would also neutralize Mr. Lysaght's comments. If we may add an implication of Mr. Tuckerman's discussion into this reply, it would appear that a hardness testing machine is an arbitrary loading mechanism containing an arbitrary measuring scale and often using for calibrating purposes an arbitrary piece of steel. To approach the goal of identical results might require the employment of the very same testing machine.

The tests were confined to very "hard" (resistant to deformation) materials in order to avoid the complexity that soft, flowable metals like copper, zinc or aluminum would introduce at the lower end of the hardness number scales. We might add, for the record, that the Vickers' impressions obtained were sharp, straight-sided and smooth, as becomes an extremely fine-grained, polycrystalline metal.

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[7] Metallurgists, Development and Research Division, The International Nickel Co., Inc., Bayonne, N. J.

Messrs. Flanders and Heyer bring in a ray of hope for improvements in hardness testing from their careful observations and rich experience. Lessening the loads or penetrator impressions in testing such complex materials as white cast irons would, of course, reduce the area integrated into the final result, but if greater consistency and accuracy is thus obtained, the practical wisdom of their recommendations is immediately applicable. However, changing the conditions of test, as well as changing the character of the irons and comparing unalloyed white iron or heat-treated cast irons with nickel-chromium cast iron, may introduce differences, as so ably pointed out by Tuckerman.

We regret that we have no answer for Mr. MacKenzie. Finally, we wish to thank the participants in the discussion and express our deep appreciation for the suggestions that had been developed.

THE PROPERTIES AND USES OF CHILLED IRON

By S. C. Massari[1]

Synopsis

It is the purpose of this paper to describe briefly the basic metallurgy of chilled iron, its technique of production, and metallographic nature.

Recent developments in the heat treatment of chilled iron with the subsequent improvement in physical properties and metallographic structure are described.

The effect of some of the generally used alloys on the properties of chilled iron is briefly discussed.

Chilled iron is one of the oldest structural materials, having been used extensively for the manufacture of the first railroad car wheels as early as the beginning of the 19th century. Similarly, for many years, both rough and finishing rolls for hot and cold working of metals have been manufactured of chilled iron, and the wearing parts of various types of crushing machines have been and still are made of the same material. The outstanding physical characteristics of chilled iron are extreme hardness and high compressive strength to resist wear from either very sharp abrasive materials or under conditions of extremely high intensity of pressure. Since chilled iron is a cast material, it lends itself unusually well for the production of intricately shaped objects which would be impossible to manufacture by other methods.

Basic Metallurgy

Technique of Production:

Molten metal as it issues from the melting furnace has all of its carbon in solution. If the metal cools slowly part of this carbon is precipitated and exists in the final casting in the form of minute flakes of graphite dispersed throughout the metal. If the metal is of proper chemical balance and instead of being poured into a sand mold is cast against a metal insert in the mold which dissipates the heat from the molten metal extremely rapidly, precipitation of carbon is prevented and when cold the casting is found to have a layer of white iron in which practically all of the carbon has been retained in the combined form instead of being precipitated as graphite. Car wheels, for example, are manufactured by casting the metal against a chiller which extends from the apex of the flange to the edge of the rim, producing a wheel which is extremely hard and resistant to wear from both rail and brake shoe in service. The white iron or chill consists essentially of iron carbide, one of the hardest constituents in a long series of iron alloys, in a matrix of pearlite. The fracture of such a chilled casting is shown in Fig. 1.

Chilled iron, in reality, consists of not one but three kinds of metal: the chill proper in which practically all of the carbon is in the combined state (largely in the form of iron carbide), the mottled iron immediately under the chill containing some graphitic or free carbon, and the interior of gray iron possessing the usual ratio of combined and graphitic carbon characteristic of ordinary gray iron castings. The greater portion of chilled iron castings manufactured today is produced from metal melted in the cupola, having a total carbon content ranging from 3.25 per cent to 3.75 per cent. In order that such an iron, when poured against a metal mold, will produce chill, it is essential that the silicon content of the iron be comparatively low. The silicon concentration must be regulated according to the quantity of total carbon in the metal, and the section thickness of the casting which is to be poured. The higher the total carbon content the lower the silicon, in order to produce a given depth of chill. Thus, it is evident that silicon is the control element which the foundryman uses for the regulation of chill depth in the final casting. Figure 1 shows the fracture of a chilled iron casting together with the approximate combined and graphitic carbon concentration in the various zones. In addition, typical Brinell, Rockwell and scleroscope hardness values are indicated for the chill, mottled, and gray iron portions of the casting.

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[1] Metallurgist, Association of Manufacturers of Chilled Car Wheels, Chicago, Ill.

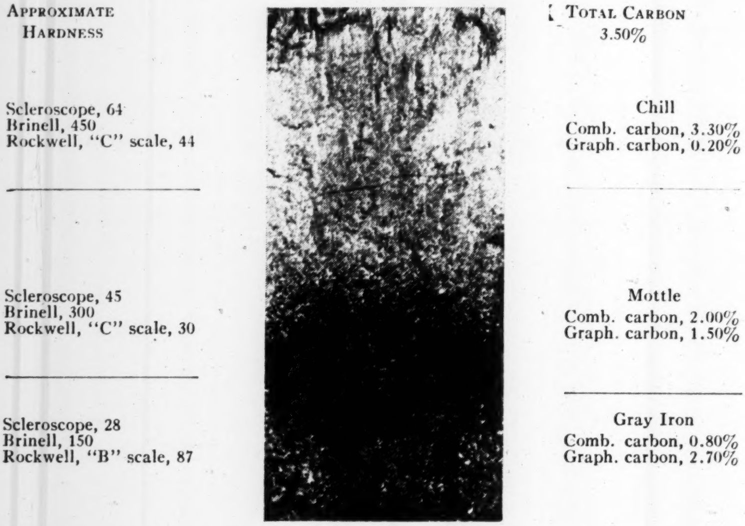

APPROXIMATE HARDNESS
Scleroscope, 64
Brinell, 450
Rockwell, "C" scale, 44
Scleroscope, 45
Brinell, 300
Rockwell, "C" scale, 30
Scleroscope, 28
Brinell, 150
Rockwell, "B" scale, 87
TOTAL CARBON 3.50%
Chill
Comb. carbon, 3.30%
Graph. carbon, 0.20%
Mottle
Comb. carbon, 2.00%
Graph. carbon, 1.50%
Gray Iron
Comb. carbon, 0.80%
Graph. carbon, 2.70%

FIG. 1.—Fracture of a Chilled Casting Showing the Approximate Combined, Graphitic Carbon Content, Hardness of the White, Mottled, and Gray Iron in the As-Cast Condition.

Effect of Total Carbon:

By controlling the total carbon content of the metal, the physical properties of the resulting chilled iron can be regulated to accommodate specific requirements in physical properties. When extreme hardness is essential and high impact strength is not necessary, a high total carbon iron can be used. If, however, a high-strength chill is required and extreme hardness is not essential, a reduction in the concentration of carbon will produce the desired result. Figure 2 shows the relation between total carbon and the scleroscope hardness of chilled iron. The results of several investigators are recorded.

The variations in test results can be accounted for by the cooling rate of the resultant casting, slower cooling tending to give slightly lower hardness values due to annealing.

Very few data are available on the tensile properties, because of the extreme hardness of chilled iron. An accurate specimen is difficult to prepare without unreasonable expenditure of time and labor. It is doubtful whether tension tests on chilled iron are generally useful inasmuch as chilled iron is rarely applied in locations where it is subjected to drastic tensile stresses. At the present time Subcommittee XIX on Chilled and White Iron Castings of the Society's Committee A-3 on Cast Iron is preparing plans for an investigation of the tensile properties by attempting to cast tension test bars in graphite molds which have been accurately machined to test specimen dimensions and thereby obviating the necessity for costly machine work. It is hoped that this investigation will yield a fund of information long desired. Up to the present time, rather meager test results indicate that the white iron has a tensile strength slightly in excess of the gray iron portions of the same casting and, quite naturally, is higher for low total carbon iron and lower for high total carbon iron, ranging from about 70,000 lb. per sq. in. to as low as 20,000 lb. per sq. in. The only marked difference in tensile properties between the white and gray portions of the same casting is a definitely higher modulus of elasticity in the chill, being about 20,000,000 lb. per sq. in. for the white as compared with approximately 15,000,000 lb. per sq. in. for the gray portions of the same iron.

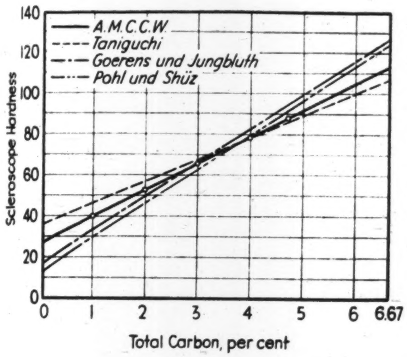


FIG. 2.—The Total Carbon Content of Chilled Iron and Its Effect on the As-Cast Scleroscope Hardness of the Chill.

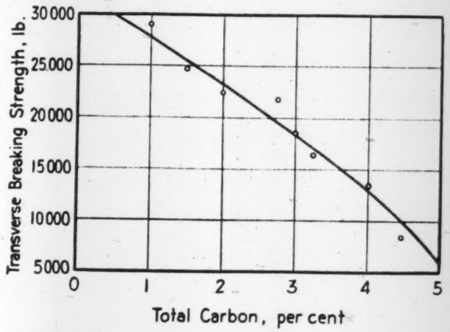


FIG. 3.—The Total Carbon Content of Chilled Iron and Its Influence on the Transverse Breaking Strength.

It is believed that a transverse test specimen cast against a chiller so as to produce white, mottled, and gray iron zones will give a much more reliable index of the quality of the iron in terms of service performance than will tension tests of each of the three different zones. Recently a series of such tests was conducted, utilizing a bar 22 in. long and having a cross-section 1½ by 2½ in. with one of the 1½ by 22-in. faces cast against a chiller. The transverse tests were made in a 100,000-lb. universal testing machine by supporting the bars on 18-in. centers, applying a concentrated load in the center and having the chill face in tension.

The effect of total carbon on the

transverse breaking strength of chilled iron is graphically shown in Fig. 3.

This investigation clearly established the relation between the total carbon concentration and the transverse breaking strength of the chilled iron. Under the conditions of these tests, the strength of the chilled iron is almost directly proportional to the reduction in total carbon.

From the foregoing it becomes apparent that if both hardness and strength are of equal importance, it is possible materially to improve the strength of the chilled iron by reducing the total carbon content without proportionately decreasing the hardness. In other words, the reduction in hardness is not so great in proportion as the improvement in strength, for a given decrease in total carbon. At least under certain conditions a low total carbon chilled iron, being softer, does not predicate less resistance to wear.

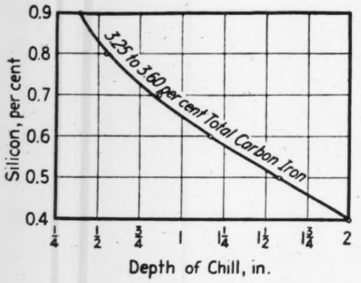


FIG. 4.—The Relation Between the Depth of Chill on Test Block and the Silicon Concentration for Chilled Irons Ranging from 3.25 to 3.60 per cent Total Carbon.

Influence of Silicon:

By controlling the silicon concentration of the iron, the foundryman is able to regulate the depth of chill in the final casting. Although the reduction in the depth of chill with increase of silicon is not a straight-line relation beyond about 0.90 per cent silicon, it is practically so when the silicon content of the iron is less than this value.

Figure 4 shows the relation between silicon and the depth of chill produced on a chill test block with an iron containing from 3.25 to 3.60 per cent total carbon, if normal equilibrium conditions exist in the molten metal. This chill test block is used by the chilled car wheel industry for control purposes, one being cast for each eight to ten wheels poured. The block is 1½ in. wide, 3 in. thick, 6 in. long, a 1½ by 6-in. face being cast against a chiller. The remainder of the mold is green sand. Iron which has been even slightly oxidized during melting may require as much as 50 per cent higher silicon content in order to produce the same depth of chill on a given casting. This condition becomes more pronounced in lower total carbon irons.

Variations in silicon from as low as 0.40 per cent to a maximum of 0.90 per cent have no apparent effect on the hardness of the chill. Likewise, the silicon concentration within these limits apparently does not influence the resistance of the chill to wear when subjected to abrasion tests. Even though silicon does not appear to influence the usual physical properties of the metal, it must be given important consideration if the chilled iron castings are to be heat treated. Silicon tends to raise the critical range of the iron and increase the rate at which the chill will be decomposed or graphitized at a given heat-treating temperature.

Microstructure of Chilled Iron:

The microstructure of chilled iron is as important a means of predicting the probable physical properties of the metal as in the case of steel. Most of the important improvements in chilled iron in recent years have been accomplished

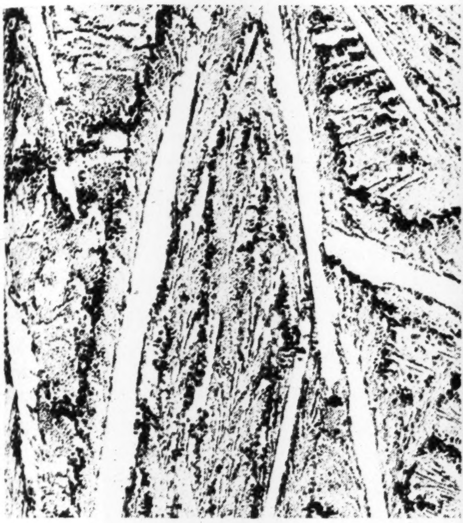


FIG. 5.—A 4.3 per cent Total Carbon Chill (× 100).

Etched with alcoholic nitric acid. The massive primary carbides are extremely coarse and extended well-defined planes of cleavage are present.

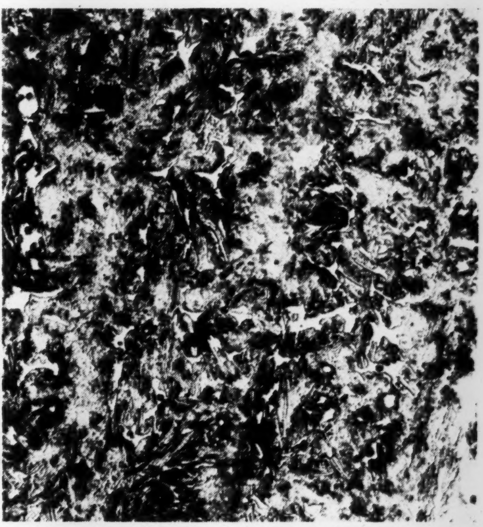


FIG. 6.—Typical Structure of a 2.50 per cent Total Carbon Chill (× 100).

Etched with alcoholic nitric acid. This metal is devoid of coarse primary carbides and planes of weakness along which failure can propagate.

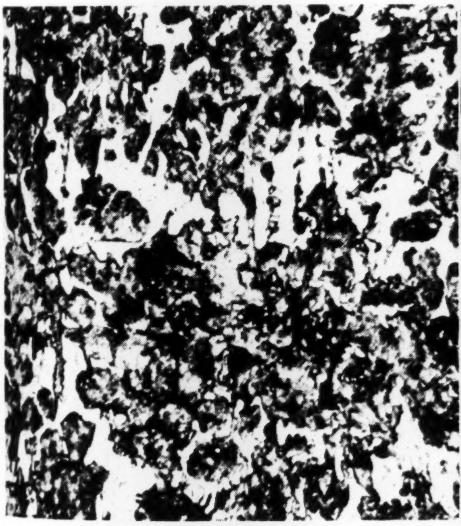


FIG. 7.—Mottled Iron Containing 3.50 per cent Total Carbon (× 100).

Etched with alcoholic nitric acid. The structure consists of primary graphite and primary cementite in a matrix of pearlite.

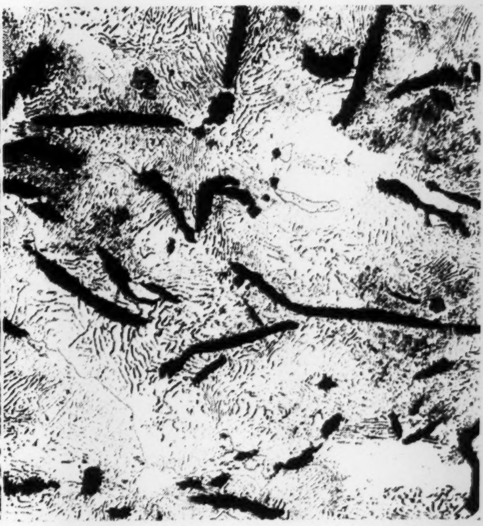


FIG. 8.—Gray Iron Portion of a Chilled Casting (× 100).

Etched with alcoholic nitric acid. The structure is typical of the usual gray iron casting, consisting of primary graphite in a matrix of lamellar pearlite.

as a result of a careful study of the microcharacter of the metal. The low transverse strength and extreme hardness of high total carbon chill is clearly understood as compared with the much higher strength and lower hardness of low total carbon iron, if the metal is examined under the microscope.

Figure 5 is typical of a high total carbon chill. The structure contains extremely coarse massive primary carbides and a highly dendritic structure with marked planes of weakness along which failure can propagate.

Similarly, and at the same magnification, Fig. 6 is typical of a relatively low total carbon chill. It is apparent that the primary carbides are much finer and uniformly dispersed. The structure is devoid of marked columnar tendencies and planes of weakness along which failure can readily progress.

The microstructure also definitely justifies the lower hardness of low total carbon chill because of the much smaller quantity of massive primary carbides which are the principle constituents contributing to the hardness of the metal. Through the use of recently developed heat-treating methods or the addition of alloys, it is possible to modify the usual structure of the matrix and materially increase the usual as-cast hardness of the metal. This will be covered more completely under "Structural Refinement." The characteristic structure of the mottled zone is recorded in Fig. 7.

The structure is composed of some primary carbides, the beginning of graphitization in the form of small clusters of graphitic carbon and a lamellar pearlitic matrix.

In Fig. 8 is shown the structure of the gray portions of the casting. With the exception of the carbon which is in solid solution in the matrix (about 0.80 per cent, the eutectoid of iron and carbon), the carbon has been precipitated in the form of graphite.

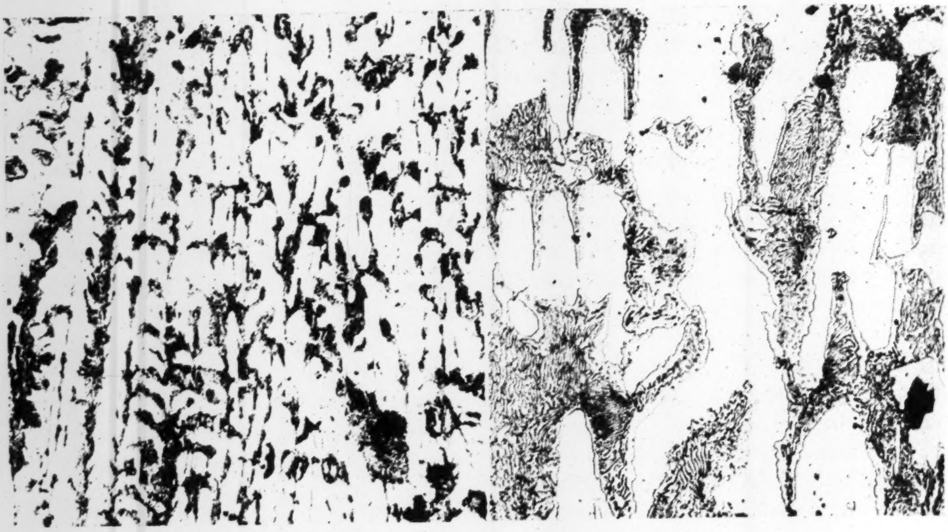
Thus, it becomes evident that improvement in the properties of chilled iron entails the modification and coincident improvement of not one, but three different kinds of metal within the same casting. The use of alloys and the development of heat-treating cycles must be directed so as to beneficially modify the entire structure simultaneously and not one to the detriment of the other.

HEAT TREATMENT

The necessity for heat treatment of chilled iron arises in a number of ways. When a casting is poured against a chiller, the surface in contact with the chiller is first to solidify, then the metal immediately under it, namely, the mottle, and finally the gray portions of the casting. Differences in the time of solidification in the various parts of the same casting result in casting stresses, and continued differences in the rate of cooling down to room temperature result in further stresses due to one part of the casting passing through the critical range before the remainder of the casting. For the sake of illustration, the chilled car wheel may be considered a typical chilled iron casting. When the wheel is poured, the chill in the flange and tread is first to solidify because of the extremely rapid dissipation of the heat from the molten metal into the chiller. Following the freezing of the metal in the tread, the plate is next to solidify and by so doing becomes integral with the tread section. Finally, the hub freezes, but instead of being free to shrink, it is restrained by the tread and plate, so that the plate is in radial tension. If the radial thickness of the hub of the wheel is assumed to be about $3\frac{1}{4}$ in. and the shrinkage $\frac{1}{8}$ in. per ft., the resulting radial strain imposed on the plate will be about 0.034 in., which will develop a tensile stress of approximately 27,000 lb. per sq. in. Under these circumstances it is not difficult to understand why a wheel that is not properly annealed will crack in the plate, since the metal has already been stressed to within 75 per cent of its ultimate strength and possesses a small reserve to resist external loads. In addition to the tensile stresses in the plate of the wheel there are also circumferential tensile stresses in the flange of the wheel. The first metal entering the mold after half the hub has been filled becomes part of the flange. Because of the rapid dissipation of heat into the chiller, and due to the comparatively small amount of metal in the flange, it is probably the first metal to solidify. Following very closely thereafter, the chilled portion of the tread becomes solid. It is quite likely that the metal in the flange has passed below the critical temperature a considerable length of time before the remainder of the tread has done so, and consequently, the abnormally high expansion, characteristic of the critical range, produces circumferential tension in the flange. The circumferential flange tension is most likely responsible for some of the small cracks which are in evidence either at the time the wheel is shaken out or when removed from the annealing pits. The infrequency with which flange checks are encountered would indicate that the flange is usually hot enough and possesses sufficient ductility to accommodate the abrupt change in volume when the tread is going through the critical range. Even though the stresses are not sufficient to cause actual rupture they may be present in varying degrees and quite naturally detract proportionately from the potential strength of the flange.

Added to the internal stresses produced by unequal cooling rates in different parts of the wheel, other stresses develop in the flange and tread section and have as their origin the difference in shrinkage between chill and gray iron. The chilled iron wheel, although being poured from metal having a uniform composition, in its finished form contains metal of widely divergent chemical analysis and physical properties. The flange and chilled portion of the tread consist of metal which has 95 per cent of its carbon in the combined state and 5 per cent in the graphitic form. Chill may essentially be considered similar to steel, since practically all of the carbon is held in solid solution and as a consequence has about twice the shrinkage of gray iron. The total shrinkage of steel and very likely chill as well is generally accepted as $\frac{1}{4}$ in. per ft. Immediately following the chill is mottled iron having 60 per cent of its carbon in the combined state and 40 per cent in the graphitic state. The remainder of the wheel is gray iron with but 25 per cent of its carbon in solid solution and 75 per cent as free graphite. The total shrinkage of gray iron is but $\frac{1}{8}$ in. per ft. and the difference in shrinkage between chill and gray iron is directly attributable to precipitation of graphite (with specific volume) which apparently overcomes about 50 per cent of the normal liquid and solid shrinkage of the metal.

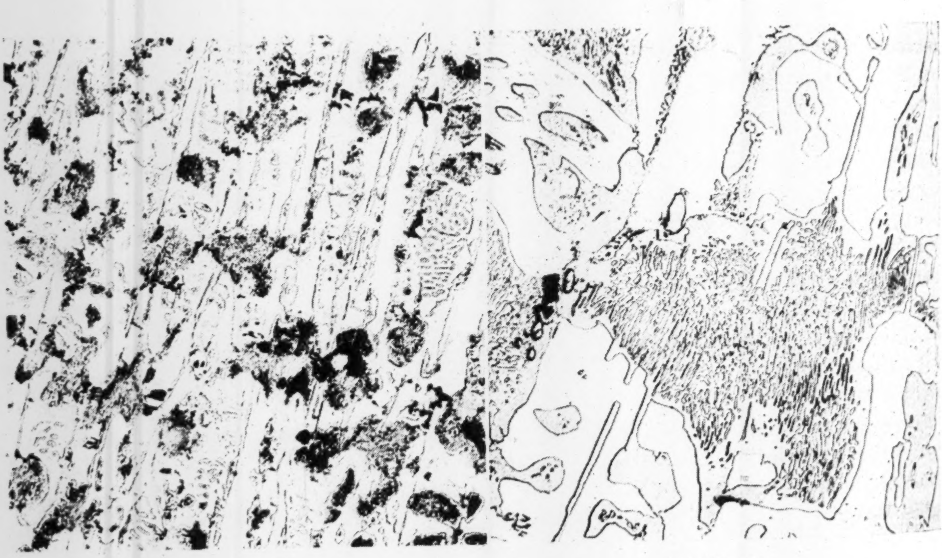
To be conservative, assume that the differential shrinkage as between mottled iron and chill is $\frac{1}{16}$ in. instead of $\frac{1}{8}$ in. per ft., since the metal immediately under the chill is mottled iron and very likely has a shrinkage intermediate between gray iron and chill. If the wheel has a chill depth of 1 in., the strain due to differential shrinkage will be $\frac{1}{16} \times \frac{1}{12}$ and will equal 0.005 in. The stress-strain diagram for wheel iron in tension reveals that the stress corresponding to this strain is approximately 30,000 lb. per sq. in. With the accepted strength of wheel iron in tension at



(a) × 100. (b) × 400.

FIG. 9.—Poorly Annealed Chilled Iron.

Etched with alcoholic nitric acid. The massive primary carbides are very coarse and the matrix is composed of distorted lamellar pearlite.



(a) × 100. (b) × 400.

FIG. 10.—Typical Structure of Well-Annealed Chill.

Practically all of the internal casting stresses have been relieved. The massive primary carbides are fine and the pearlite lamellae have straightened and assumed their normal form.

37,000 lb. per sq. in. there is only a 20 per cent surplus to prevent rupture or resist some external stress.

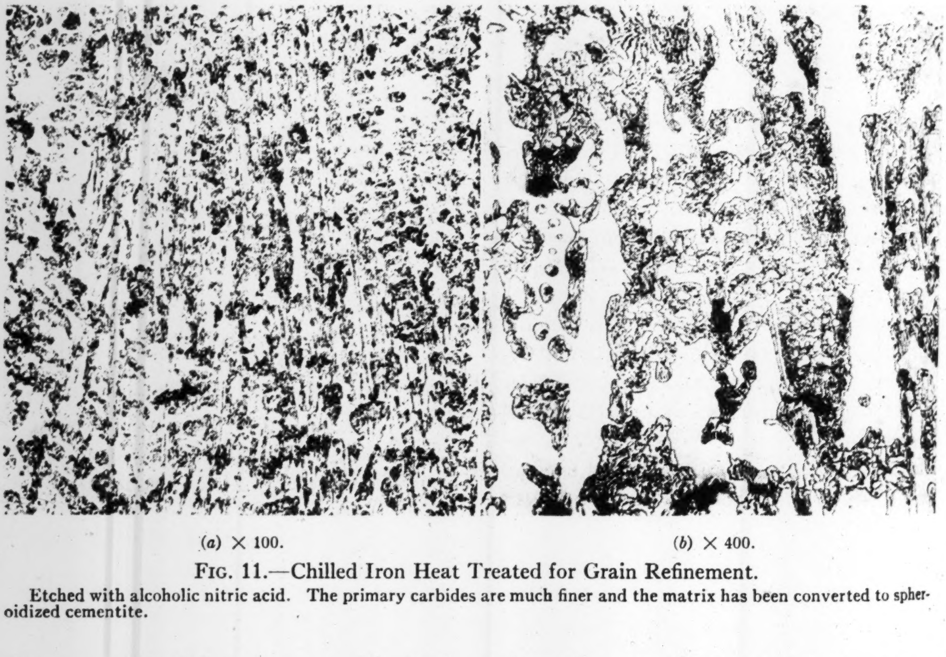
From the foregoing, it is apparent that a chilled casting which is symmetrical in design, such as a wheel or roll, when shaken from the mold may contain internal stresses closely approaching the ultimate strength of the metal. A casting such as this must be relieved of internal stresses by heat treatment if the casting is to successfully resist service stresses imposed upon it.

Detection of Internal Stresses:

Although not a direct means for the detection of residual internal stresses, there are certain microstructures which are typical of either improperly heat-treated metal containing internal stresses or material which has received proper annealing and is free from stresses. Faulty annealing is the result of an insufficiently high equalizing temperature, lack of sufficient time at temperature or too rapid a cooling rate as the metal passes through its critical range. Specific heat-treating temperatures followed by definitely controlled cooling rates result in specific metallographic structures in the metal. The presence of these structures can be used as a means for establishing the probable heat treatment to which the metal had been subjected. Furthermore, distorted rather than clearly defined structures are indicative of internal stress, the normal crystallizing forces having been opposed to such an extent that the normal arrangement could not develop. Photomicrographs of poorly heat-treated chill, high in internal stress and giving a very poor test is shown in Fig. 9 (*a*) and (*b*). The structure is coarse and highly dendritic. The white structureless constituent is massive primary cementite, produced in the initial formation of the chill when the wheel was cast. The constituent between the cementite is lamellar pearlite. Careful examination will reveal that the lamellae instead of being straight and regular are distorted and tend to be knotty due to the presence of residual internal stress. Similarly, photomicrographs of well heat-treated chill are shown in Fig. 10 (*a*) and (*b*) at the same magnifications. The important difference between this metal and that portrayed in Fig. 9 is the smaller amount of massive primary cementite, the absence of coarse columnar dendrites and straightening of the pearlite lamellae, the normal crystallographic form of this constituent.

Structural Refinement:

Heat treatment of chilled iron to improve the physical properties is of primary importance. In fact it is even more important because the metal receives no working either hot or cold between the time it is cast and the time when the casting is ready to ship to the customer. The only manner in which the as-cast structure can be modified in a chilled casting is by heat treatment or the use of alloys coupled with heat treatment. All metals when solidifying from the liquid state form dendritic grains. The chill portion of a chilled casting is almost free to develop as it chooses, particularly in a direction perpendicular to the surface of the chiller. Under the microscope the dendritic structure of the chill is very evident and more completely developed in weaker or high total carbon irons. Failure of chilled iron castings due to impact blows struck in a plain parallel with the grain of the metal is much more prevalent in coarse-grain metals than in fine-grain ones. Although it is highly essential that the as-cast structure of the metal be as fine as possible, considerable refinement and improvement in the physical properties of the chilled iron can be accomplished by properly devised and accurately controlled heat-treating cycles. Research has well established the practicability of heat treating chilled iron at relatively high temperatures to accomplish the partial re-solution of coarse primary carbides with their subsequent re-precipitation on cooling so as to produce a fine and more uniform grain structure in the chill. Such metal shows a marked improvement in physical properties with but a very small loss in hardness or loss in wear resistance. For example: a chilled iron heat treated at a temperature of 1500 F. for 18 hr. and then permitted to cool at 10 deg. per hr. to 1200 F. before removal from the furnace exhibits a microstructure as shown in Fig. 11(*a*) and (*b*).



(*a*) × 100. (*b*) × 400.

FIG. 11.—Chilled Iron Heat Treated for Grain Refinement.

Etched with alcoholic nitric acid. The primary carbides are much finer and the matrix has been converted to spheroidized cementite.

The massive primary carbides are very much finer, most of the highly developed dendritic structure has been eliminated, the matrix has been converted to spheroidized cementite, and no evidence of graphitization of the chill has occurred. Similar improvement can be accomplished at a temperature of 1600 F. for 8 hr. Chilled transverse test specimens, of the dimensions already described, when heat treated at either 1500 or 1600 F. for the time specified, show an increase in strength of about 40 per cent over the as-cast condition. The best results have been obtained when the metal is heat treated at temperatures ranging from 1500 to 1600 F. It is very essential, however, that the metal be not exposed to these elevated temperatures for sufficient time to graphitize the primary carbides but instead heat treatment must only be continued to the beginning of incipient graphitization. A long series of tests involving chilled iron specimens poured from metal containing 3.25 to 3.60 per cent total carbon, 0.50 to 0.55 per cent silicon, 0.55 to 0.60 per cent manganese, 0.12 to 0.14 per cent sulfur and 0.30 to 0.35 per cent phosphorus, when heat treated at 1500, 1550, and 1600 F. yielded the results shown in Figs. 12, 13 and 14.

This investigation definitely directs attention to the necessity for accurately controlling the length of time the castings are held at the elevated temperatures in order to prevent the graphitization of the chill with a coincident marked decrease in hardness. The accuracy of control becomes increasingly more important as the heat-treating temperature becomes higher. At a temperature of 1500 F. the castings may be held for a maximum of 20 hr.; at 1550 F. for a maximum time of 9 hr.; and at 1600 F. a maximum time of about 7 hr. Within these limits the strength of the metal and resistance to impact can be increased from 30 to 50 per cent. The time limits given apply only to metal having a silicon content of 0.50 to 0.55 per cent; higher silicon values will naturally increase the rate of graphitization and less time at temperature will result in greater decrease in hardness of the chill.

ALLOY CHILLED IRON

The addition of alloys to chilled iron involves a careful consideration of the physical properties desired and the effect of these alloys on the normal iron-carbon-silicon equilibrium. The value of the alloy is completely lost unless it is added to a base metal of proper chemical balance and unless it has been melted under conditions conducive to producing high quality metal. Alloys cannot improve a poor base metal, or, rather, alloys are not capable of converting a fundamentally poor metal into a good one. Alloys primarily tend to refine the as-cast grain structure of the metal and improve its response to heat treatment. The alloys serve not only to enhance the properties of the chilled metal but also those of the mottled and gray iron as well.

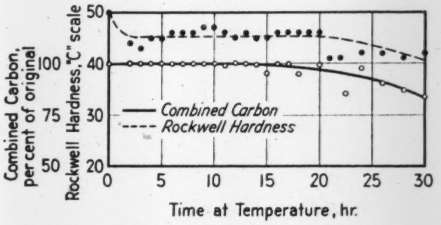


FIG. 12.—The Effect of Time on the Hardness of the Chill and Its Graphitic Carbon Content when Chilled Iron Is Heat Treated at 1500 F.

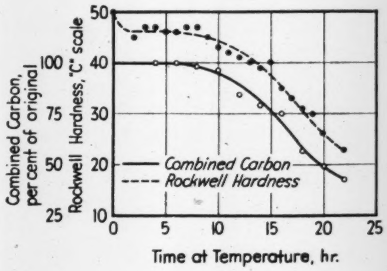


FIG. 13.—The Effect of Time on the Hardness of the Chill and Its Graphitic Carbon Content when Chilled Iron Is Heat Treated at 1550 F.

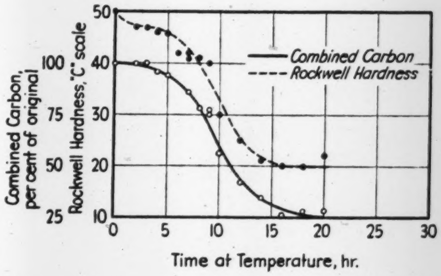


FIG. 14.—The Effect of Time on the Hardness of the Chill and Its Graphitic Carbon Content when Chilled Iron Is Heat Treated at 1600 F.

With the exception of tungsten, alloys added to chilled iron may be classified according to whether or not they increase or decrease the depth of chill on a given casting, when poured from a given base metal. Alloys tending to increase the depth of chill when added in a given base iron are: chromium, manganese, vanadium, and molybdenum. Elements which tend to reduce the depth of chill in varying amounts are: carbon, silicon, aluminum, titanium, nickel, copper, cobalt, and zirconium. The comparative influence of these elements on the depth of chill on a chilled iron roll is shown in Figs. 15 and 16.[2]

Elements that Reduce the Depth of Chill:

Carbon.—Carbon exerts a very pronounced influence on the depth of chill produced in a given casting and is almost as effective in this respect as silicon. Although not in general use as yet, comparatively small quantities of amorphous graphite added to the ladle, just prior to filling preparatory to pouring a mold, will substantially reduce the depth of chill on the resulting casting. The reaction with the metal is probably two-fold: first, one of deoxidization, and, second, but more important, it supplies the seeds for subsequent graphite precipitation during solidification.

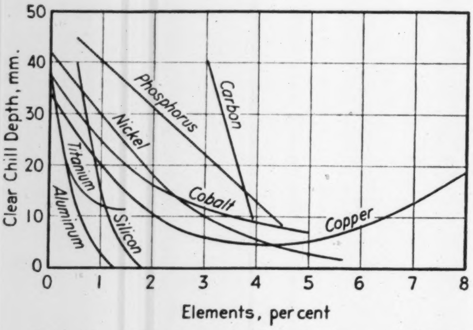


FIG. 15.—The Comparative Influence of Various Elements on the Chill Depth of a Chilled Iron Roll. Elements which decrease the depth of chill.

Silicon.—Silicon is by far the cheapest and most widely used element for the control of chill depth. As already pointed out, its effect is not uniform in all concentrations but is practically so when the silicon is less than 0.90 per cent. This is clearly portrayed in Fig. 4. Ferrosilicon additions made to the ladle, so-called late additions, are about twice as effective in reducing the depth of chill as an equal quantity of silicon added to the furnace charge. Silicon has no apparent effect on the grain structure of the chill, except that as the silicon is increased the depth of chill becomes less and coincident with it, the columnar grain structure is minimized. This effect is, however, not the direct effect of the silicon on the grain growth but rather due to the lesser depth of chill the dendrites are unable to grow as long. Within the limits of chill formation on a casting, silicon does not appear to influence either the hardness of the chill or its wear resistance when subjected to abrasion tests. Silicon raises the Ac range approximately 53 deg. Fahr. for each 1 per cent increase in silicon, and as a consequence if the castings are to be heat treated the silicon concentration must be considered.

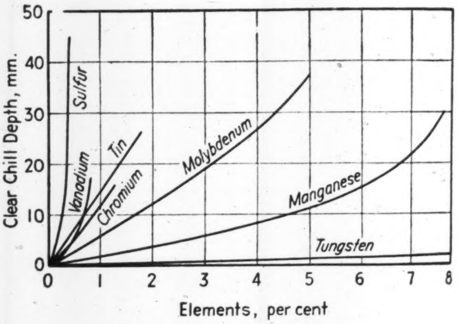


FIG. 16.—The Comparative Influence of Various Elements on the Chill Depth of a Chilled Iron Roll. Elements which increase the depth of chill.

Aluminum.—Similar to silicon, aluminum is a powerful graphitizer even in very small concentrations. Small quantities of aluminum may be used as ladle additions to serve as a deoxidizer and chill reducer, but quantities in excess of approximately 0.1 per cent of aluminum should not be used. Aluminum added in excess of the quantity required for deoxidization of the iron is prone to produce pin holes in the casting. Its ability to stabilize the equilibrium of the metal is probably its greatest attribute and aluminum should not be used as an alloying material in the ordinary sense but only as a scavenger.

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[2] Kohei Taniguchi, "Study of Alloy Chilled Iron Rolls," *Japan Nickel Review*, Vol. I, No. 1, April, 1933, p. 32.

Titanium.—Titanium is a powerful graphitizer and hence will tend to reduce the depth of chill if added to a given base metal. This alloy also has a strong affinity for both oxygen and nitrogen and hence may prove useful as a deoxidizer to improve the fluidity and stability of oxidized metal. In quantities less than 1½ per cent, it has no apparent effect on the hardness of the chill.

Cobalt.—Cobalt reduces the depth of chill when added to a given base metal about one-fourth as much as an equal quantity of silicon. Probably because of its cost and the fact that other elements can be used for a similar purpose this element is not used commercially as an alloying material in chilled iron.

Nickel.—One of the most widely used alloys in chilled iron is nickel. This element is not normally used alone; it is used to better advantage in combination with other elements. Its wide application as an alloy in chilled iron is due to its beneficial influence on the physical properties of metal combined with its relatively moderate cost. About four times as much nickel as silicon is required to accomplish the same reduction in depth of chill. As the concentration of nickel is increased, the hardness gradually increases from 60 to 70 scleroscope for an unalloyed iron, to approximately 90 when a concentration of 4½ per cent nickel is reached. Beyond this value the hardness again gradually diminishes. When nickel is added to chilled iron in increasing quantitites, the matrix of the chill changes from pearlite to sorbite, to troostite, and finally to martensite when 4½ per cent nickel has been attained. If the nickel concentration exceeds about 4½ per cent, the matrix is converted to austenite instead of martensite and the resulting hardness gradually diminishes due to the lower hardness of austenite as compared with martensite. Nickel refines the grain structure of the chill and gray portions of the casting as well. When nickel is used as an alloying material it is usually in combination with chromium in the ratio of about 2½ to 3 parts of nickel to one part of chromium so as to maintain a constant depth of chill in a given base metal.

Copper.—In concentrations under 4 per cent, copper gradually diminishes the depth of chill but in excess of this value tends gradually to increase the chill depth. In concentrations under 3 per cent it tends to slightly increase the hardness. Like nickel, it is usually added to iron in combination with chromium in order to maintain a constant depth of chill. A fairly large tonnage of chilled castings is being made with 0.75 to 1.00 per cent copper. It is stated that this alloy addition makes it possible to control the depth of chill more accurately by stabilizing the behavior of the metal and increases the fluidity of the iron.

Zirconium.—Zirconium is most useful as a deoxidizer and exerts a mild graphitizing action on the chilled iron. Its principal value is exhibited when added to the ladle prior to pouring. In concentrations less than 0.1 per cent, it serves to stabilize the chilling tendency of the iron without apparently affecting other physical properties of the metal.

Elements that Increase the Depth of Chill:

Chromium.—Chromium is the cheapest and most useful element for increasing the depth of chill. About three-quarters as much chromium will counteract a given quantity of silicon and thus, chromium affords the foundryman a means for saving a heat having an excess of silicon which would otherwise produce castings having insufficient chill. In concentrations up to a maximum of 4 per cent, the hardness of the chilled iron is increased approximately ten points on the scleroscope scale, but higher concentrations yield a decreased hardness. Chromium tends to coarsen the grain structure of the chill, broaden the mottled zone and close the grain of the gray iron. Chromium raises the Ac critical temperature approximately 120 deg. Fahr. for each 1 per cent of chromium. Hence, iron which is to be heat treated must be heated to a higher temperature if a re-solution of primary carbides is desired. No comprehensive or authentic data are available as to whether chromium improves the wear resistance of chilled iron.

Manganese.—Although manganese is seldom used as an alloying material, it is one of the most useful elements normally present in chilled iron. By reacting with the sulfur present, the detrimental effects of this latter element are completely eliminated. If the manganese concentration is twice the sulfur content of the iron plus 0.30 per cent, the ill effects of sulfur are completely eliminated. Many attempts have been made to condemn chilled iron containing in excess of 0.12 to 0.14 per cent sulfur, but an investigation involving iron containing as much as 0.20 per cent sulfur disclosed that if present with sufficient manganese, it possesses physical properties equal to irons containing less than 0.10 per cent sulfur. When manganese is added to chilled iron in increasing quantities, it tends to lower the depth of chill until the demands of the sulfur have been satisfied. In excess of this concentration it increases the depth of chill about one-fourth as fast as chromium. As manganese is increased the hardness of the chill gradually increases, attaining a maximum value of about 85 scleroscope with a concentration of approximately 7 per cent. High concentrations of manganese increase the shrinkage of the iron and accentuate the columnar structure of the chill. High chill depth produced by manganese is very brittle and highly susceptible to temperature gradients or thermal shock.

Vanadium.—Because of its potency, vanadium in concentrations as low as 0.25 per cent exercises a beneficial effect on the structure of the chill. This element is a potent carbide stabilizer and even in low concentrations produces a marked increase in the depth of the chill. Vanadium refines the structure of the chill, minimizes coarse columnar grain structure, and improves the physical properties of the mottled and gray iron. The high cost of vanadium is probably its chief disadvantage.

Molybdenum.—Molybdenum gradually increases the depth of chill, being about one-third as effective as chromium in this respect. In concentrations of 0.50 per cent or above, it refines the grain structure of the chill and mottled iron. It also reduces the particle size of the graphite in the gray portions of the casting and accomplishes an increase of about 20 per cent in tensile strength when 0.50 to 0.75 per cent of molybdenum is added to a given base iron.

Alloy Combinations:

Nickel-Chromium.—When high superficial hardness is essential such as for the manufacture of highly polished chilled-iron finishing rolls, a scleroscope hardness of over 80 can be attained in chilled iron. With a total carbon content of 4 per cent or greater, the chill will have a hardness of approximately 80 scleroscope, but the metal will be extremely coarse and possess low resistance to impact failure. The same hardness can be attained by the use of 4 to 6 per cent of nickel and 1 to $2\frac{1}{2}$ per cent of chromium. This metal will be fine grained, high in strength, and show excellent resistance to impact. With the proper ratio of nickel and chromium added to a high-grade base iron, scleroscope hardness values as high as 90 may be attained. With a nickel to chromium ratio of approximately three to one the hardness of the chill will be increased without affecting the depth of chill. The chill in this metal will consist of fine massive primary cementite in a matrix of martensite if the iron contains 4 to 6 per cent nickel and 1 to $2\frac{1}{2}$ per cent of chromium. To attain further toughness 0.25 to 0.35 per cent of molybdenum or 0.35 per cent of vanadium is added. Such castings lend themselves well to heat treatment and if heated to 1650 to 1700 F., held 1 hr. per in. of thickness, quenched in oil and then drawn at 400 F. the chill will be toughened due to a partial re-solution of the massive primary carbides. If slightly lower hardness can be tolerated, the castings may be slowly heated to 1600 F., held for sufficient time to attain uniform temperature and then permitted to cool slowly in the furnace. Such a treatment further toughens the metal and lends itself better to large castings which might crack during quenching because of drastic temperature gradients during this operation.

Copper-Chromium.—If low cost is essential, and an increase of 15 to 20 per cent in transverse breaking strength is sufficient, the addition of $1\frac{1}{2}$ per cent of copper and 0.5 per cent of chromium together with ample silicon to compensate for the increase in chill depth, due to the chromium, will accomplish the desired results. This alloy combination will have a modest refining action on the as-cast structure of the chill and mottled iron.

Chromium-Molybdenum.—The as-cast columnar structure of the chill can be materially refined by the addition of molybdenum and chromium. Even with concentrations as low as 0.50 per cent molybdenum and 0.20 per cent chromium, the grain structure of the chill is finer. Probably economically the greatest benefit from molybdenum is not attained until 0.75 per cent molybdenum and 0.20 per cent chromium are added to a given base metal. This alloy addition will produce an extremely fine-grained metal substantially improved in physical properties.

USES OF CHILLED IRON

The practical application of chilled iron is very diversified. It finds its use where resistance to wear and deformation under high intensities of pressure are the essential requisites. This can be even more readily appreciated when it is realized that the compressive strength of unalloyed chilled iron is in excess of 200,000 lb. per sq. in. and the hardness over 60 scleroscope. Chilled railroad car wheels probably represent the largest tonnage of any single item manufactured from chilled iron. For the past ten years over two million wheels annually have been produced of chilled iron, representing a yearly tonnage of approximately 750,000 tons. The large annual tonnage of this material justifies an extensive research to develop the full potential properties of the metal. The next largest single item is very likely chilled iron rolls for crushing grain or ore, and the rough and finish rolling of metals. In addition to these, a long list of items such as heavy-duty castings for coal crushing, grinding mill liners, grinding balls, cement grinding machinery, jaw crusher plates, stamp mill parts, etc., are quite generally made of chilled iron. Frequently, the liners in sludge pumps and the wearing plates in chutes handling abrasive materials are also made of chilled iron.

All railroad car wheels and, in all probability, a substantial percentage of other chilled iron castings are made of unalloyed iron. For specific applications, however, the introduction of alloys into chilled iron materially enhances their performance, so that there is a distinct economical advantage in their use. For example, roll crusher shells used to resist wear and abrasion when crushing ore, gravel or stone, when made of chilled iron containing 4.50 per cent nickel and 1.50 per cent chromium give 50 per cent longer service than if the same parts are made of more costly manganese steel and almost three times the service of an unalloyed chilled iron. The same alloy chilled iron gave about twice the service of manganese steel when used for jaw crusher plates, also crushing ore, gravel or stone. Space does not permit giving concrete examples of the many applications of chilled iron other than to make mention of the variety of economical uses for this material and to cite a few specific examples.

DISCUSSION

Mr. H. Bornstein.[1]—I want to compliment the author. I think he has done a fine job in stimulating thought and research on the subject of white and chilled cast iron.

Being in the farm implement business, I am a little disturbed that he did not put down farm implement castings when he mentioned the uses of chilled iron castings. We do use quite a few thousand tons of chilled and white iron castings every year for such items as plow-shares, mold boards and various parts, bearings, etc.

Mr. Massari has mentioned the effect of carbon. We have found in our work, particularly on such castings as bearings where we use white iron, that the carbon content is important. We formerly used a white iron produced primarily for malleable castings, which iron has a total carbon content in the neighborhood of 2.4 per cent, and we found that the wear resistance was not particularly good. The Brinell hardness is in the neighborhood of 340 and it was desired to increase both the hardness and the wear resistance. We did this by using a cupola white iron, increasing the carbon content to approximately 3.5 per cent.

Mr. Massari mentioned the matter of internal stress, particularly in wheels. I can sympathize with Mr. Massari in this. Among other things, we make chilled castings, such as chilled sprockets, and we have had field failures on items of that type where the stress in the field appeared to be very low in comparison with the apparent strength of the material. For example, one casting was designed for a stress of about 3500 lb. per sq. in. and we still had failure. On measuring the internal stresses of these castings, we found that the internal stress in some instances ran as high as 28,000 lb. per sq. in. which was pretty nearly the strength of the material.

Mr. R. S. MacPherran[2] (*presented in written form*).—Mr. Massari's paper is a most concise résumé and I only regret that space did not permit more detailed description of their cupola operation and testing of metal in the ladle.

It would be most interesting to see the stress diagram to which the author refers and also his preferred composition and desired microstructure for wheels. There is no doubt that this information, though referring primarily to wheels, would apply almost equally to other chilled castings.

The author mentions a dendritic structure. It would be interesting to know if they make any effort to avoid this structure and if they find it undesirable in service.

The President (*Mr. A. E. White*[3]).—When we realize that but a few short years ago there were those who felt that the use of cast iron had reached its zenith, and when we now realize the further extensions which are being made in the field of cast iron and the developments which have taken place, we can realize the great progress which has taken place. It is not long ago that we thought of cast iron in terms of chemical composition and physical properties only. No one, except in the strictly scientific sense, thought of its metallographic structure. Today there is as much emphasis on metallographic structure as on chemical composition for cast irons for certain specific service. The author has forcibly brought this matter out in his paper.

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[1] Director of Laboratories, Deere and Co., Moline, Ill.

[2] Chief Chemist, Allis-Chalmers Manufacturing Co., West Allis, Wis.

[3] Director of Department of Engineering Research, and Professor of Metallurgical Engineering, University of Michigan, Ann Arbor, Mich.

MR. S. C. SPALDING.[4]—I was surprised to notice in the first figure shown that the scleroscope hardnesses were shown from 100 to 130. That seemed rather startling to me. I did not suppose that any metal would run as high as that. I wondered whether it were possible to get such readings.

MR. S. C. MASSARI[5] (*author's closure by letter*).—I wish to thank Mr. Bornstein for calling attention to the applications of chilled iron in the farm implement industry. The uses enumerated in the paper represent merely some of the typical cases where chilled iron is employed because of its inherent characteristics. Referring to the relationship between total carbon, strength and hardness, if an improved chilled iron is desired and only moderate loss in hardness can be tolerated, it is possible to produce a 3 per cent total carbon iron that will have a Brinell hardness ranging from 390 to 400. I should like to cite results of a test made on car wheels to emphasize the value of stress relief. This test is made by supporting the wheel flange at two equi-distant points in the testing machine and applying a static load on the hub. Wheels containing supposedly substantial internal stresses will fail at a total load less than 100,000 lb. When given a stress relief heat treatment, the breaking load will quite consistently run 125,000 lb., and if given a grain refinement treatment, increase to nearly 175,000 lb.

Mr. MacPherran raises the question of cupola operation. It is necessary in the chilled car wheel industry to maintain a silicon control of plus or minus 0.05 per cent. This is not a matter of choice, but absolutely vital to the production of a saleable product. An investigation is in progress at the present time that we hope will accomplish much closer cupola control. The desirable microstructure for wheels shows partial refinement of the as-cast primary carbide structure with a spheroidized cementite matrix. There is no way at present of controlling the dendritic structure. The stress-strain diagram referred to by Mr. MacPherran is simply a conventional stress-strain diagram obtained by plotting data of elastic properties of iron. It has been obtained on gray, mottled, and chilled iron in order to have a definite knowledge of the relative elasticity and strength of the three materials.

Mr. Spalding brings up the point of high scleroscope hardness. This is primarily an extrapolation of data. The author has never observed the hardness of chilled iron in excess of about ninety, and that attained in rather highly alloyed or very high total carbon iron. On one occasion, in making experimental heats in a high-frequency induction furnace by superheating to very high temperatures and introducing Atcheson graphite, the author was able to increase the total carbon content of the metal to very nearly five per cent. This material in the cast condition, probably in part due to strain hardness, had a scleroscope hardness of about ninety. Such hardness cannot be attained with any degree of regularity, nor would the author recommend the production of an iron of this type with the expectation that it would stand any substantial external stresses.

In conclusion the author wishes to thank the President, Mr. White, for his comments.

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[4] Metallurgical Engineer, The American Brass Co., Waterbury, Conn.

[5] Metallurgist, Association of Manufacturers of Chilled Car Wheels, Chicago, Ill.

EFFECT OF SIZE AND TYPE OF SPECIMEN ON THE TORSIONAL PROPERTIES OF CAST IRON

By Jasper O. Draffin[1] and W. L. Collins[1]

Synopsis

Tests were made on 48 machined specimens, 24 from each of two heats of cast iron, the material being a part of the bars from which the tension specimens reported on last year[2] were taken. The specimens were (1) solid, $\frac{3}{4}$ in. and 1 in. in diameter, and (2) hollow, 1 in. in outside diameter with wall thicknesses of $\frac{1}{8}$ in., $\frac{1}{4}$ in. and $\frac{11}{32}$ in. Strain measurements were made on all specimens. The torsional properties of the iron were found to be practically the same for both diameters of the solid specimens. For the hollow specimens the maximum unit strain and the modulus of rupture were found to decrease as the wall thickness decreased. The modulus of rupture obtained with a wall thickness of $\frac{1}{8}$ in., or a ratio of wall thickness to outside radius, $\frac{t}{r}$, of 0.243, was approximately equal to the tensile strength of the cast iron when tested in direct tension.

A year ago the authors presented data[2] on the tensile strength of cast iron, dealing principally with a means of finding accurately the tensile strength. As originally planned, the tests as outlined provided specimens for making torsion and bending tests as well as tension tests. The intention was to make a series of tests through the entire range of tension, torsion, compression and bending, on the same kind of iron, hoping it might be possible to correlate the strengths of the cast iron under different kinds of stress. The tests in torsion are reported here together with a summary of the tension tests previously reported. No specific effort is here made to correlate the strengths in tension and torsion.

Materials and Test Specimens

All specimens were machined from 3 by 3 by 56-in. bars which were cast in a vertical position in the Machine Laboratories of the University of Illinois. The castings were poured in two heats, designated as series F and series A. Both series were of similar composition, the mix consisting of one-third scrap steel, one-third scrap cast iron and one-third new pig iron. The composition was estimated by the foundry to be about as follows: total carbon 3.45 per cent, silicon 1.60 per cent, manganese 0.55 per cent, sulfur 0.09 per cent and phosphorus 0.45 per cent. The typical microstructure of the iron, from pieces cut from the rectangular ends of torsion specimens, is shown in Fig. 1. Specimens for microscopic examination were cut across the entire cross-section of the 3 by 3-in. bars and these were polished and etched. While not shown in this paper, these sections showed a uniform structure over the entire width of the bars.

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[1] Professor, and Associate, respectively, Department of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[2] J. O. Draffin and W. L. Collins, "The Tensile Strength of Cast Iron," *Proceedings*, Am. Soc. Testing Mats., Vol. 37, Part II, p. 88 (1937).

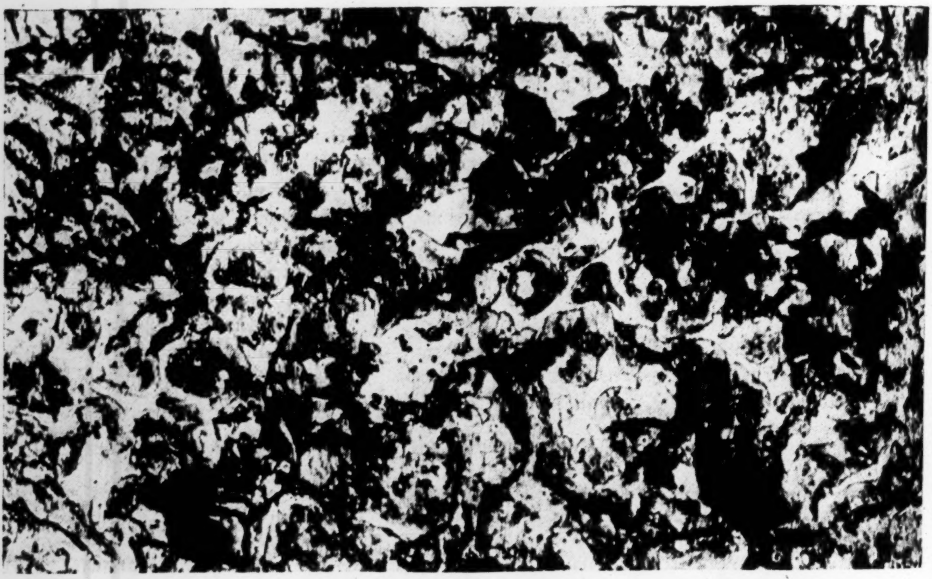
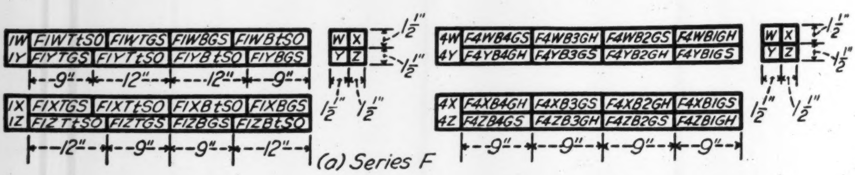
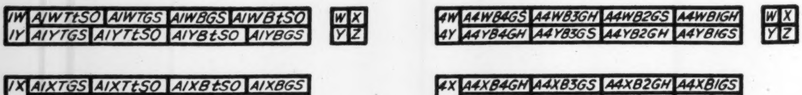


FIG. 1.—Photomicrograph of Cast Iron, Etched with 4 per cent Nital (× 100).


1W
1Y
F1WTtSO
F1WTGS
F1WBGS
F1WBtSO
F1YTGS
F1YTtSO
F1YBtSO
F1YBGS
9"
12"
12"
9"
1X
1Z
F1XTGS
F1XTtSO
F1XBtSO
F1XBGS
F1ZTtSO
F1ZTGS
F1ZBGS
F1ZBtSO
12"
9"
9"
12"
W
X
Y
Z
1½"
1½"
1½"
1½"
4W
4Y
F4WB4GS
F4WB3GH
F4WB2GS
F4WB1GH
F4YB4GH
F4YB3GS
F4YB2GH
F4YB1GS
4X
4Z
F4XB4GH
F4XB3GS
F4XB2GH
F4XB1GS
F4ZB4GS
F4ZB3GH
F4ZB2GS
F4ZB1GH
9"
9"
9"
9"
W
X
Y
Z
1½"
1½"
1½"
1½"

(a) Series F


1W
1Y
A1WTtSO
A1WTGS
A1WBGS
A1WBtSO
A1YTGS
A1YTtSO
A1YBtSO
A1YBGS
1X
1Z
A1XTGS
A1XTtSO
A1XBtSO
A1XBGS
A1ZTtSO
A1ZTGS
A1ZBGS
A1ZBtSO
W
X
Y
Z
4W
4Y
A4WB4GS
A4WB3GH
A4WB2GS
A4WB1GH
A4YB4GH
A4YB3GS
A4YB2GH
A4YB1GS
4X
4Z
A4XB4GH
A4XB3GS
A4XB2GH
A4XB1GS
A4ZB4GS
A4ZB3GH
A4ZB2GS
A4ZB1GH
W
X
Y
Z

(b) Series A

FIG. 2.—Position of Specimens in Casting.

The location in the casting of the specimens tested in tension and torsion for series F and series A are shown in Fig. 2. The tension specimens were solid, 0.80 in. in diameter, with threaded ends. The longitudinal strains were measured by means of a strain gage over an 8-in. gage length at six gage lines spaced at equal intervals around the circumference. The holes for the points of the strain gage were drilled in projecting collars machined on the specimen. Because of the collars, as shown in the previous paper,[2] the holes for the strain gage had no weakening effect on the specimens.

The torsion specimens were of the general dimensions and types shown in

Fig. 3. The hollow specimens were all drilled and, except for those with the $\frac{5}{16}$-in. holes, were reamed to secure a smooth inner surface. The testing was done in a pendulum torsion machine having a capacity of 10,000 in-lb. The angle of twist was measured over a 5-in. gage length by means of the torsion indicator shown in Fig. 4.

Data from Tests

Table I gives a summary of the test results in tension for both series F and series A. Tables II and III give detailed data for the tests in torsion; specimen numbers ending in "S" designate solid specimens while those ending in "H" designate hollow specimens. Figure 5 shows typical twisting moment - deformation curves for 1-in. and $\frac{3}{4}$-in. solid bars. Figure 6 shows typical twisting moment - deformation curves for solid and hollow bars having 1-in. outside diameters and different wall thicknesses; the upper part of Fig. 6 contains the enlarged portions of the curves. All specimens tested in torsion failed in tension with the helical fracture characteristic of cast iron, and the position of the fracture was not influenced by the indentations made by the screws of the attached torsion indicator or by changes in section of the specimen.

Table I.—Tensile Strength of Cast Iron.

Series and Bar Number	Number of Tests	Tensile Strength, lb. per sq. in.	Tensile Unit Strain, in. per inch	Modulus of Elasticity, lb. per sq. in.
F1........	8	max. 31 400	0.00698	17 000 000
		min. 29 900	0.00543	14 000 000
		avg. 30 800	0.00638	15 100 000
F2........	16	max. 28 900	0.00696	14 700 000
		min. 25 000	0.00406	11 000 000
		avg. 27 700	0.00588	13 000 000
F4........	No tension tests			
A1........	8	max 29 300	0.00760	13 100 000
		min. 25 800	0.00525	11 400 000
		avg. 28 000	0.00686	12 400 000
A4........	No tension tests			

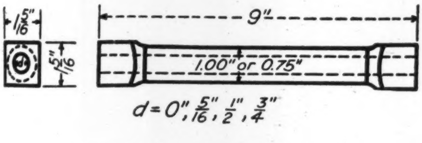


Fig. 3.—Torsion Specimen.

The modulus of elasticity was determined by drawing a straight line through the mean of the plotted points below 1200 to 1500 in-lb. thus giving comparative results. In computing the data from the curves it was assumed that the tensile unit stress equals the shearing unit stress and that the tensile unit strain equals one-half the shearing unit strain.

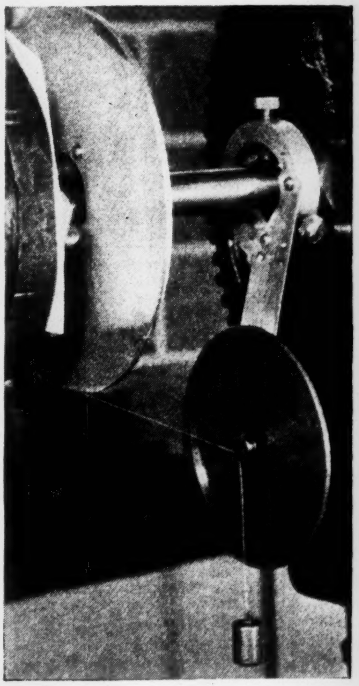


Fig. 4.—Torsion Indicator Used to Measure Angle of Twist.

Brinell hardness tests were made on 43 of the 48 specimens tested in torsion

TABLE II.—DATA ON SOLID AND HOLLOW TORSION SPECIMENS.

Series F.

Specimen Number	Outside Diameter, in.	Inside Diameter, in.	Ratio, Wall Thickness to Outside Radius	Maximum Twisting Moment, in-lb.	Modulus of Rupture, lb. per sq. in.	Modulus of Elasticity, lb. per sq. in.	Maximum Shearing Unit Strain, in. per inch	Maximum Tensile Unit Strain, in. per inch
F1YTGS	1.000	....	1.000	7900	40 200	5 740 000	0.0335	0.01675
F1WTGS	0.999	....	1.000	8300	42 500	6 500 000	0.0399	0.01995
F1ZTGS	1.000	....	1.000	8000	40 800	7 540 000	0.0370	0.01850
F1XTGS	1.001	....	1.000	7650	39 000	6 350 000	0.0282	0.01410
F1XBGS	1.000	....	1.000	8260	42 200	7 900 000	0.0387	0.01935
F1ZBGS	0.999	....	1.000	8040	41 100	7 470 000	0.0375	0.01875
F1WBGS	1.000	....	1.000	8100	41 200	6 720 000	0.0395	0.01975
F1YBGS	0.995	....	1.000	8030	40 900	7 380 000	0.0425	0.02125
Average........				8030	40 900	6 950 000	0.0371	0.01855
F4WB4GS	0.999	....	1.000	7300	37 300	6 300 000	0.03295	0.01647
F4ZB4GS	1.002	....	1.000	7850	39 900	6 580 000	0.03800	0.01900
F4YB4GH	0.999	0.509	0.490	6120	33 600	6 430 000	0.02460	0.01230
F4XB4GH	0.999	0.753	0.246	3450	26 000	5 900 000	0.01350	0.00675
F4XB3GS	0.999	....	1.000	7000	35 800	6 080 000	0.02730	0.01365
F4YB3GS	1.000	....	1.000	7810	39 700	6 180 000	0.04000	0.02000
F4WB3GH	0.999	0.507	0.492	6040	33 100	6 830 000	0.02375	0.01187
F4ZB3GH	0.995	0.507	0.490	5770	31 900	6 230 000	0.20500	0.01025
F4WB2GS	1.000	....	1.000	7600	38 600	7 500 000	0.03650	0.01825
F4ZB2GS	1.000	....	1.000	7790	39 600	6 520 000	0.03900	0.01950
F4YB2GH	1.002	0.508	0.494	6140	33 200	5 950 000	0.02400	0.01200
F4XB2GH	1.000	0.752	0.248	3520	26 400	5 840 000	0.01450	0.00725
F4XB1GS	1.000	....	1.000	7700	39 200	6 600 000	0.03750	0.01875
F4YB1GS	1.000	....	1.000	7790	39 600	6 370 000	0.03700	0.01850
F4WB1GH	1.000	0.752	0.248	3250	24 300	5 820 000	0.00990	0.00495
F4ZB1GH	1.000	0.754	0.246	3830	28 800	5 900 000	0.01650	0.00825
				AVERAGES				
Solid........			1.000	7530	38 700	6 520 000	0.03598	0.01799
¼-in. wall........			0.492	6020	33 000	6 380 000	0.02321	0.01160
⅛-in. wall........			0.247	3510	26 400	5 870 000	0.01360	0.00680

TABLE III.—DATA ON SOLID AND HOLLOW TORSION SPECIMENS.

Series A.

Specimen Number	Outside Diameter, in.	Inside Diameter, in.	Ratio, Wall Thickness to Outside Radius	Maximum Twisting Moment, in-lb.	Modulus of Rupture, lb. per sq. in.	Modulus of Elasticity, lb. per sq. in.	Maximum Shearing Unit Strain, in. per inch	Maximum Tensile Unit Strain, in. per inch
A1XTGS	1.000	....	1.000	8030	40 900	5 800 000	0.0449	0.0225
A1YTGS	0.749	....	1.000	3380	41 000	6 670 000	0.0456	0.0228
A1WTGS	1.000	....	1.000	7450	38 000	5 800 000	0.0362	0.0181
A1ZTGS	0.750	....	1.000	3340	40 300	5 800 000	0.0446	0.0223
A1ZBGS	1.000	....	1.000	7720	39 300	6 080 000	0.0440	0.0220
A1WBGS	0.748	....	1.000	3230	39 300	5 870 000	0.0438	0.0219
A1YBGS	0.998	....	1.000	7600	39 000	6 020 000	0.0452	0.0226
A1XBGS	0.748	....	1.000	3170	38 600	5 900 000	0.0413	0.0206
Average 1 in........				7700	39 300	5 930 000	0.0426	0.0213
Average ¾ in........				3280	39 800	6 060 000	0.0438	0.0219
A4WB4GS	0.998	....	1.000	7650	39 200	6 370 000	0.0373	0.01865
A4ZB4GH	0.999	0.313	0.685	6820	35 300	5 850 000	0.0260	0.01300
A4XB4GH	0.999	0.511	0.488	5760	31 600	5 810 000	0.0210	0.01050
A4YB4GH	0.996	0.756	0.244	3670	27 900	5 820 000	0.0165	0.00825
A4YB3GS	0.999	....	1.000	7660	39 200	6 130 000	0.0390	0.01950
A4XB3GH	1.000	0.311	0.688	7120	36 600	5 630 000	0.0335	0.01675
A4WB3GH	0.999	0.511	0.488	6050	33 200	5 820 000	0.0250	0.01250
A4ZB3GH	1.000	0.754	0.246	3620	27 200	5 510 000	0.0155	0.00775
A4ZB2GS	1.000	....	1.000	7790	39 600	5 970 000	0.0440	0.02200
A4WB2GH	0.997	0.311	0.688	6820	35 400	6 520 000	0.02975	0.01487
A4YB2GH	1.000	0.508	0.492	5960	32 500	5 980 000	0.0270	0.01350
A4XB2GH	1.000	0.756	0.244	3710	28 100	5 840 000	0.01825	0.00913
A4XB1GS	0.998	....	1.000	7380	37 900	5 800 000	0.0378	0.01890
A4YB1GH	1.000	0.311	0.690	6900	35 500	5 760 000	0.03175	0.01587
A4ZB1GH	0.999	0.511	0.488	6040	33 100	6 230 000	0.0270	0.01350
A4WB1GH	0.996	0.758	0.243	3650	28 000	5 770 000	0.0180	0.00900
				AVERAGES				
Solid........			1.000	7620	39 000	6 070 000	0.0395	0.01980
11/32-in. wall........			0.688	6910	35 700	5 940 000	0.03025	0.01512
¼-in. wall........			0.489	5950	32 600	5 960 000	0.0250	0.01250
⅛-in. wall........			0.244	3660	27 800	5 740 000	0.0176	0.00853

readings being taken on four faces of the rectangular ends for about one-half of the specimens. The maximum and minimum readings were 197 and 163. The variation between faces was no greater than between specimens. Taking an average of all specimens, one reading on each specimen, the Brinell number was 183.

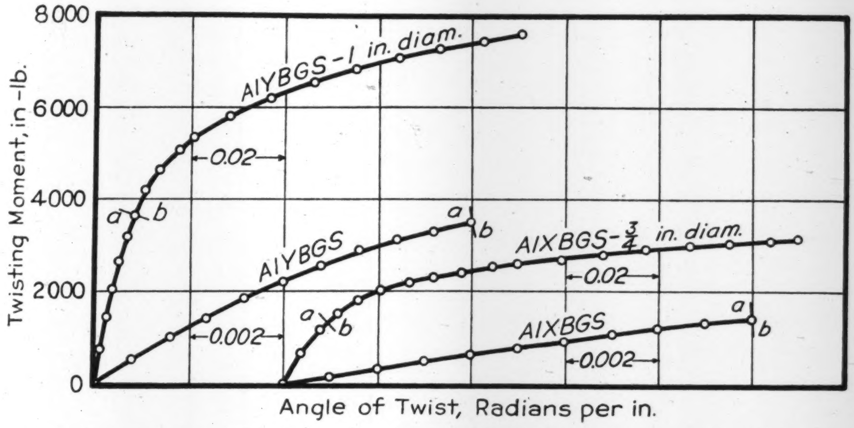


FIG. 5.—Typical Twisting Moment - Angle of Twist Diagrams for Solid Specimens.

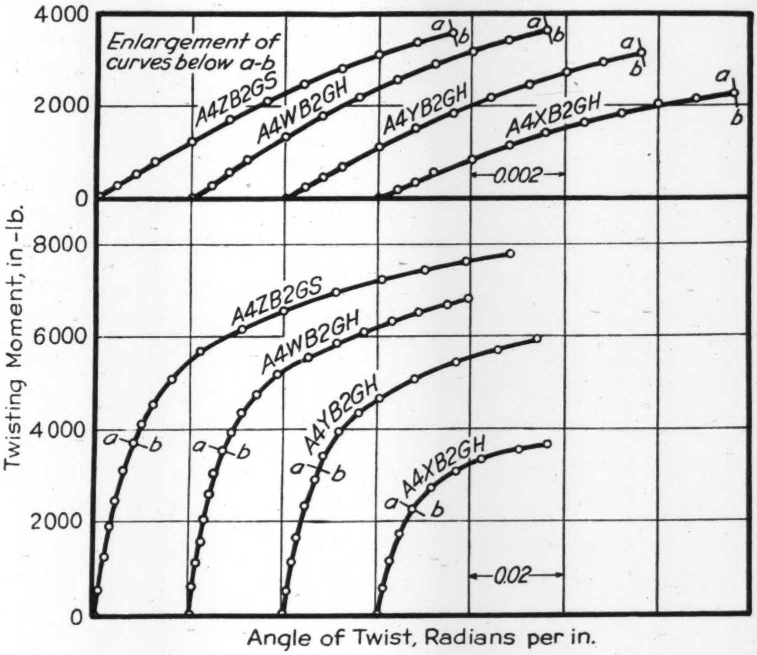


FIG. 6.—Typical Twisting Moment - Angle of Twist Diagrams for Solid and Hollow Specimens Having 1.00-in. Outside Diameter.

ANALYSIS OF DATA

The two principal topics discussed in this series of tests are how the torsional properties of cast iron, such as modulus of rupture, modulus of elasticity and the maximum unit strain vary (*a*) with the diameter of the solid specimen, and (*b*) with the wall thickness of the hollow specimen.

Effect of Diameter.—Two diameters were used, $\frac{3}{4}$ in. and 1 in., all taken from bar No. 1 of series A. Since both sizes of specimens were machined from the same relative position in the bar, there was little effect of size of casting, a conclusion which is amply borne out by the tension tests made on solid and hollow specimens cut from the same portion of the bar. Consequently any significant variation in the properties, if such be found, may be attributed to the effect of the diameter. From Table III it is seen that the average modulus of rupture for the two sizes of specimens is approximately the same, showing that the diameter has little or no effect on the strength of the specimens tested. The same is true for the modulus of elasticity and the maximum unit strain. In other words, given a cast iron of the same quality throughout the cross-section, the torsional properties of the material, of the size and quality of solid circular specimens tested, is not affected by the diameter of the specimens.

Effect of Thickness of Walls.—If the stress-strain curve for any material in shear were a straight line to failure, the equation

$$S = \frac{Tc}{J}$$

where S = the unit stress in pounds per square inch,

T = the twisting moment in inch-pounds,

c = the outside radius of the specimen in inches, and

J = the polar moment of inertia in inches^4

would hold true to failure for any circular section. But where the stress-strain curve departs from a straight line, the resisting moment actually developed is greater than the equation indicates, provided the value used for S in the equation is the actual shearing strength of the material (or the tensile strength for a brittle material like cast iron). This being true, the question arises as to the resistance developed when some of the inner portion of the cylinder is removed, or how much of the material must be removed in order to have a torsion test give the true strength of the material. Comparative tests of solid and hollow specimens of steel[3] and of aluminum alloys[4] have been made, but the authors

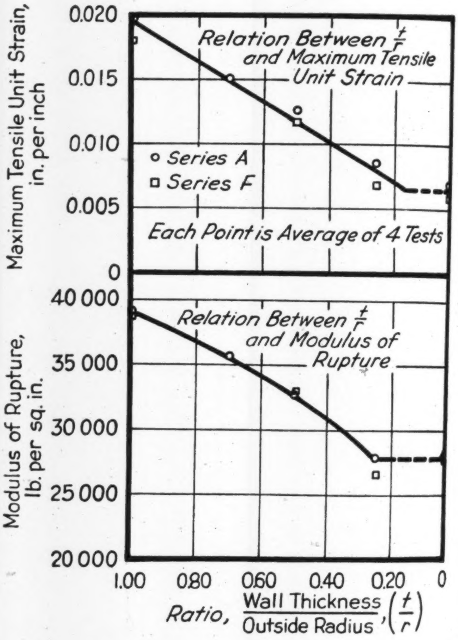


FIG. 7.—Curves Showing the Relationship in Torsion Between Modulus of Rupture and $\frac{t}{r}$ and Between Maximum Tensile Unit Strain and $\frac{t}{r}$.

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[3] F. B. Seely and W. J. Putnam, "The Relation Between the Elastic Strengths of Steel in Tension, Compression and Shear," *Bulletin No. 115*, Engineering Experiment Station, University of Illinois (1919).

[4] R. L. Templin and R. L. Moore, "Specimens for Torsion Tests of Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part II, p. 534 (1930).

are not aware of similar tests on solid and hollow specimens of cast iron.

In Fig. 7 data are plotted showing the relationship between $\frac{t}{r}$ and the modulus of rupture and between $\frac{t}{r}$ and the maximum tensile unit strain. The test values of these properties from both series of tests agree with each other as closely as can be expected.

The modulus of rupture - $\frac{t}{r}$ curve shows that the modulus of rupture decreases as the value of $\frac{t}{r}$ decreases until $\frac{t}{r}$ reaches a value of 0.243. At this value of $\frac{t}{r}$ the shearing strength developed, computed by $S = \frac{Tc}{J}$, is equal to the actual tensile strength of the cast iron as determined by direct tension tests, the values of which are plotted on the graphs for $\frac{t}{r}$ equal zero. While no tests were made with $\frac{t}{r}$ less than 0.243, it is reasonable to expect the modulus of rupture to remain constant at values of $\frac{t}{r}$ less than 0.243, provided there are no defects in the iron.

Similar to the curve for modulus of rupture, the maximum unit strain - $\frac{t}{r}$ curve shows a decrease in the maximum unit strain as the thickness of the wall is reduced. The reduction in the tensile unit strain, from that found with a solid specimen, to the value found in direct tension is reached (by extrapolation) at a value for $\frac{t}{r}$ of 0.15. The change in the value of the maximum tensile unit strain developed is significant because the solid specimens in torsion develop a tensile unit strain over three times as great as that developed in direct tension, 0.020 in. per inch in torsion and 0.006 in. per inch in tension, though all specimens tested failed by tension. It seems that cast iron is capable of considerable plastic deformation provided there is a core of low stressed material to "back up" the more highly stressed portion. This transfer of load from the outer to the inner portion of a member subjected to torsion is the principal factor which raises the modulus of rupture in torsion above the tensile strength in direct tension. Further tests on the effect of transfer of load from the higher to the lower stressed portions of members are to be made later. Considerable analytical study has been made of the relation between the tensile strength and the modulus of rupture, but further tests, some of which are now under way, are needed before satisfactory conclusions can be reached.

CONCLUSIONS

Specimens from two heats of cast iron of similar composition were tested in tension and torsion. The results obtained for each type of test were in close agreement.

For solid specimens in torsion the modulus of rupture, modulus of elasticity and maximum tensile unit strain were practically the same for diameters of 1 in. and $\frac{3}{4}$ in.

For hollow specimens in torsion, with walls $\frac{1}{8}$ in. thick, or a $\frac{t}{r}$ of 0.243, the modulus of rupture decreased from about 39,000 lb. per sq. in. for the solid specimens to about 27,000 lb. per sq. in., the latter of which is the strength of the cast iron in direct tension.

For hollow specimens in torsion, the maximum tensile unit strain decreased from about 0.020 in. per inch in the solid ones to 0.0076 in. per inch for a $\frac{t}{r}$ of 0.243, the maximum tensile unit strain in direct tension being about 0.006 in. per inch.

The reduction in the tensile unit strain, from that found with a solid specimen, to the value found in direct tension tests is reached (by extrapolation) at a value for $\frac{t}{r}$ of 0.15.

Acknowledgment:

Acknowledgment is made to Prof. C. H. Casberg and J. F. Wooddell of the Machine Laboratories of the University of Illinois for casting the bars used for the tests and also to two Senior students in the College of Engineering who assisted in the work, C. S. Sandler, who made most of the tests in torsion and plotted the data, and to J. A. Nachowitz who prepared the photomicrographs.

DISCUSSION

Mr. A. H. Dierker[1] (*presented in written form*).—Messrs. Draffin and Collins are to be congratulated on working in a worthwhile but rather neglected field. Any real contribution, such as this, to our rather meager knowledge of the strength and elastic properties of cast iron is welcome indeed.

There can be little criticism of the paper with regard to the careful preparation of the specimens, the painstaking methods used in making the tests, and the conclusions drawn. The data given, however, would seem to be more valuable if an actual analysis of the various specimens had been made. An estimated analysis always leaves a reasonable doubt as to its accuracy. One might also question certain assumptions regarding the uniformity of the specimens.

In an investigation[2] made some years ago by the writer, small specimens were cut from a large heavy section casting. Testing of the small specimens indicated that they were not uniform over their cross-section. Microscopic examination confirmed our suspicions that the side of the specimens that had been close to the outside of the casting from which it was cut showed a somewhat different microstructure from the side that had been toward the interior of the casting. The rather large differences in Brinell hardness (197 to 163) found by the authors would indicate the possible lack of uniformity in the physical structure of the metal in individual specimens. Any lack of uniformity would not, of course, show up as readily in tests of a relatively soft iron (of the type used by the authors) as it would in tests of harder, higher strength iron.

Mr. J. T. MacKenzie[3].—I think the results of the torsion test substantiate some of our thinking on the transverse test and explain some of the results on the span loading test. One thing I would, however, still question is what the authors call uniformity. Take the structure of the square which they have used for the tension test and the torsion test, assuming that the material varies uniformly from edge to center, any specimen that you would machine symmetrically out of that corner would have the same average properties as the average properties of that quarter. Now, as long as they stick to torsion and tension, I think they will find that any specimen whether a small bar of one size or a hollow bar of another size or a solid bar of any size, will be the same. I understand they are planning to investigate compressive strength (and I certainly hope they do because we need some data on compression). I fear they will find that the inside of the bar will yield at a very much faster rate than the outside.

Mr. W. R. Osgood[4] (*presented in written form*).—In discussing the lower diagram of Fig. 7, the authors state that "it is reasonable to expect the modulus of rupture to remain constant at values of $\frac{t}{r}$ less than 0.243." This statement seems to be the result of an oversight, for there can hardly be any question but that for sufficiently low values of $\frac{t}{r}$ the specimens would fail by buckling at stresses well below the tensile strength. It would be interesting, therefore, and perhaps of importance to follow the curve experimentally further to the right, at least until failure occurs by buckling. The curve may run horizontally for some distance from $\frac{t}{r} = 0.243$ and then drop when the failure is no longer a tensile failure, or it may continue its downward course from $\frac{t}{r} = 0.243$. If the latter, further light may be shed on the phenomenon of failure—or further questions raised!

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[1] Research Engineer, Engineering Experiment Station, The Ohio State University, Columbus, Ohio.

[2] A. H. Dierker, "The Effect of Size of Specimen on the Strength and Elastic Properties of Cast Iron," *Bulletin No. 72*, Engineering Experiment Station, The Ohio State University (1932).

[3] Chief Chemist, American Cast Iron Pipe Co., Birmingham, Ala.

[4] Materials Engineer, National Bureau of Standards, Washington, D. C.

The other point has to do with the method of plotting. Two bars, A and F, were used, with different properties (Table I). It is reasonable to expect to find these properties reflected in the modulus of rupture, the specimens with high tensile strength, for example, showing higher values of the modulus of rupture. It is suggested that a more accurate graphical representation of the variation of modulus of rupture with thickness-radius ratio may be obtained by plotting modulus of rupture divided by tensile strength against $\frac{t}{r}$, or possibly even against the product of $\frac{t}{r}$ and some nondimensional function of the mechanical properties, such as tensile strength and modulus of elasticity. Nondimensional plots of this kind have been found to be extremely useful in several investigations.[5]

MR. R. L. MOORE[6] (*presented in written form*).—The torsion tests described by Messrs. Draffin and Collins on cast iron are of particular interest to the writer in that they parallel a somewhat similar series of tests on wrought aluminum alloys described before this Society in 1930.[7] The principal difference in the results obtained is that tensile failures were obtained in all the torsion tests on the cast iron, while shearing or buckling failures were obtained on the aluminum.

In the analysis of the results obtained on the hollow tubes, the authors state that since the modulus of rupture for a $\frac{t}{r}$ ratio of 0.243 was equal to the tensile strength of the cast iron, as determined by the direct tension tests, "it is reasonable to expect the modulus of rupture to remain constant at values of $\frac{t}{r}$ less than 0.243, provided there are no defects in the iron." Accordingly the modulus of rupture $\frac{t}{r}$ curve in Fig. 7 has been shown as a dotted horizontal line between the limits of the tests and $\frac{t}{r} = 0.$

It is unfortunate that the authors did not carry their tests further as one would logically expect modulus of rupture values to continue to decrease with decreasing $\frac{t}{r}$. Buckling or instability failures may occur in either cast or wrought materials when the proportions of the specimens are such that the critical buckling stress[8] falls below the strength of the material in tension or shear. Therefore it is believed that the modulus of rupture - $\frac{t}{r}$ curve in Fig. 7 should be drawn to pass through the point of zero stress at $\frac{t}{r} = 0$.

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[5] See, for example: Ambrose H. Stang Walter Ramberg and Goldie Back, "Torsion Tests of Tubes," *Report No. 601*, Nat. Advisory Committee for Aeronautics, Washington, D. C. (1937); also W. R. Osgood, "Column Strength of Tubes Elastically Restrained Against Rotation at the Ends," *Report No. 615*, Nat. Advisory Committee for Aeronautics, Washington, D. C. (1938).

[6] Research Structural Engineer, Aluminum Research Laboratories, Aluminum Company of America, New Kensington, Pa.

[7] R. L. Templin and R. L. Moore, "Specimens for Torsion Tests of Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part II, p. 534 (1930).

[8] S. Timoshenko, "Theory of Elastic Stability," Article 90, p. 480, McGraw-Hill Book Co., Inc., New York City (1936).

MR. W. M. LANSFORD[9] (*by letter*).—The authors have presented the results of a very interesting series of tests in this paper. However, the writer believes a valuable addition to it could be made by, (*a*) testing specimens having smaller values of $\frac{t}{r}$ than 0.243 and, (*b*) testing specimens in which t and r are both larger than those used by the authors. Tests of specimens having values of $\frac{t}{r}$ less than 0.243 would tend to establish more certainly the lower portion of the curve shown in Fig. 7. It would be particularly valuable in establishing a general law of action of brittle materials to know how large t could be and have the value of $\frac{t}{r} = 0.243$ be the criterion to judge the true tensile stress. For specimens having large values of t, and having $\frac{t}{r} = 0.243$, the area over which the shearing stress would need to be uniformly distributed might be too large for that shearing stress to equal the tensile stress.

MESSRS. JASPER O. DRAFFIN[10] AND W. L. COLLINS[10] (*authors' closure by letter*).—The authors desire to thank all who have discussed this paper; a number of valuable suggestions were made. In view of the comments of Mr. MacKenzie and Mr. Dierker, additional information on the structure of the cast iron is presented in the accompanying Figs. 1, 2 and 3.

Photomicrographs of specimens from casting F2 cut at intervals (center, outside and two points equally spaced between) along a line connecting the centers of two opposite sides of the cross-section, are shown in the accompanying Fig. 1. No marked variation in the structure is revealed when etched in a saturated solution of ammonium persulfate and magnified 20 diameters even though the specimens came from the top of a casting just below a portion containing blow-holes.

The entire cross-section of casting F5, at the mid-point of the length, is shown in the accompanying Fig. 2. The etching medium was a saturated solution of ammonium persulfate and the photograph is 1.25 times natural size. The dark streak in the lower left-hand corner is apparently due to uneven drying of the alcohol wash and the dark spot near the center is due to an indentation caused by dropping the piece. The fine-grained structure shown appears to be quite uniform over the entire cross-section.

When an etching solution consisting of 2 parts of sulfuric acid, 1 part hydrochloric acid and 3 parts of water, heated to just below the boiling point, was used and the same specimen shown in Fig. 2 immersed in it for 30 min., the structure shown in the accompanying Fig. 3 was observed. This figure is also 1.25 times natural size. A coarse-grained dendritic structure is brought out by this etch and it is evident that there is some difference in the structure of the iron between the outside and the center of the casting. However, considering the photomicrographs as well as the manner of selection of the specimens for the tension and torsion tests, it is felt that there was not sufficient nonuniformity across the section to have much effect on the results of the tests. Since the compression tests have not been made and since the tests on a high-strength iron have just started it is not known whether or not this slight variation across the section will seriously affect the results of the tests yet to be made.

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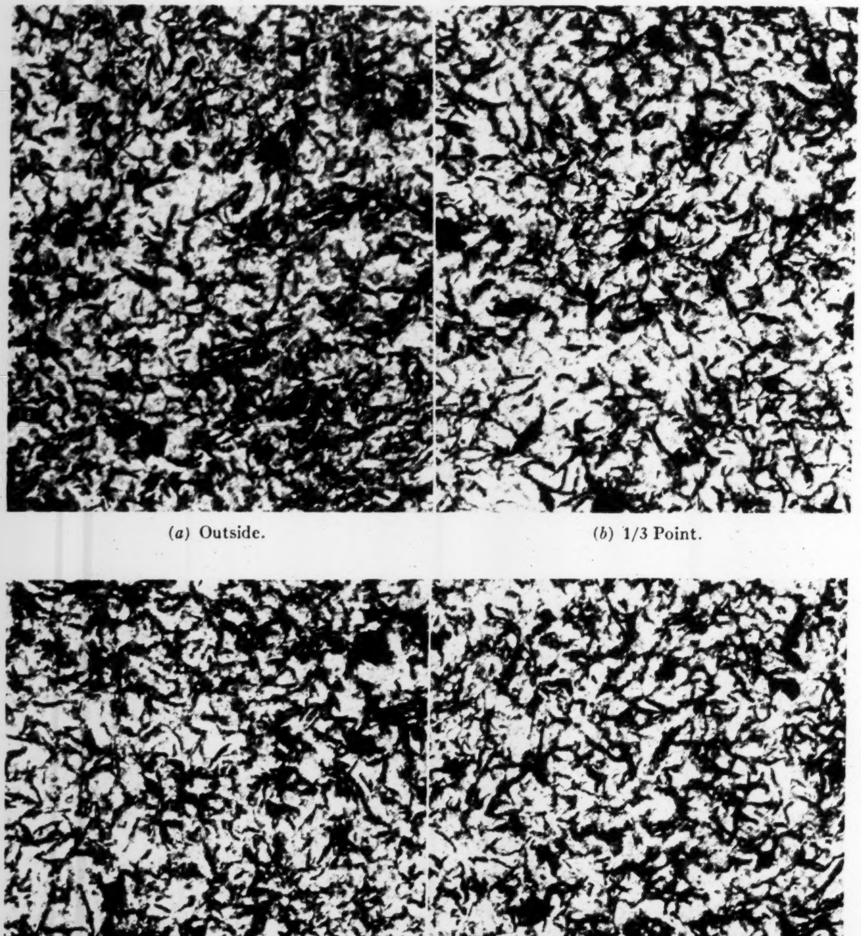
[9] Assistant Professor of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[10] Professor, and Associate, respectively, Department of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.


(a) Outside. (b) 1/3 Point.

(c) 2/3 Point. (d) Center.

FIG. 1.—Structure of Casting F2. From outside to inside of casting (× 20). Etched with saturated solution of ammonium persulfate.

The assumed uniformity is also borne out by an additional hardness study of the slab of material used for Figs. 2 and 3. Rockwell numbers are used

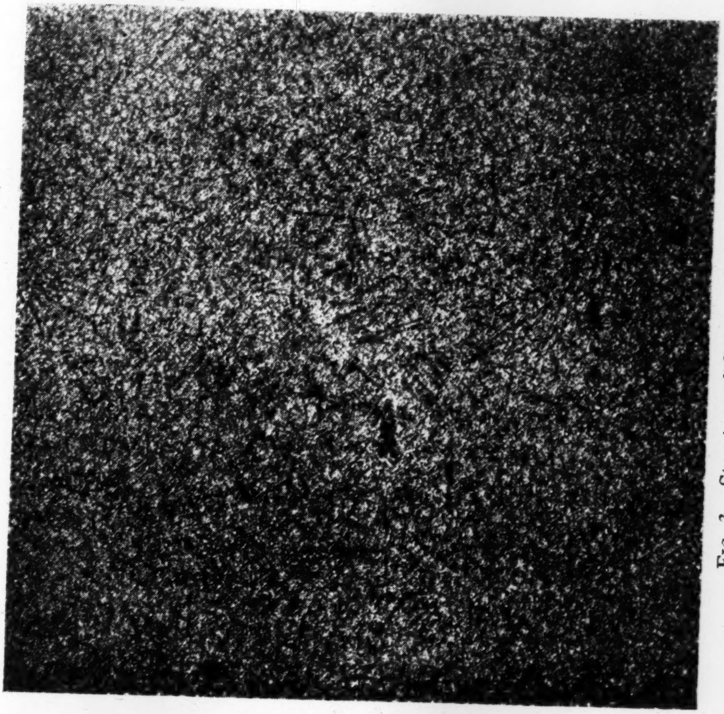


FIG. 3.—Structure of Casting F5 (× 1.25).
Etched with 2 parts H_2SO_4, 1 part HCl, and 3 parts H_2O.

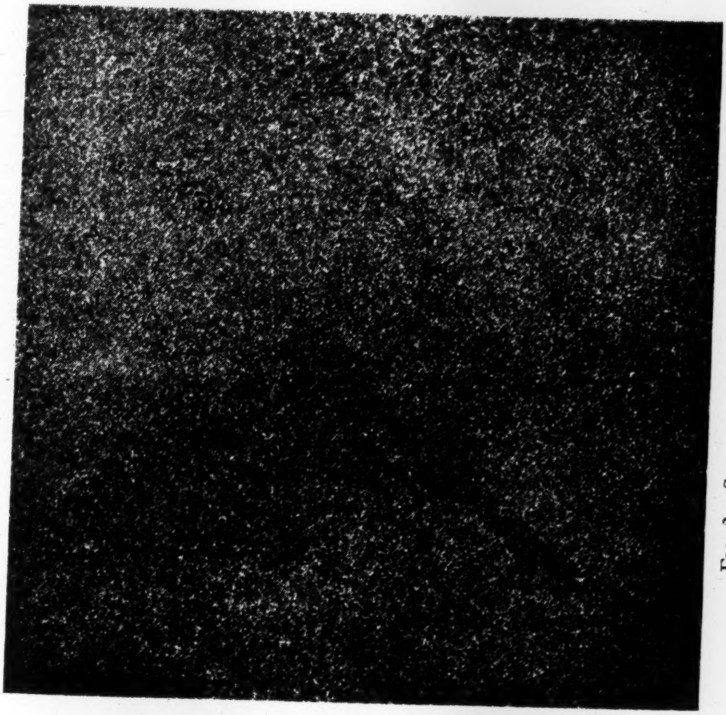


FIG. 2.—Structure of Casting F5 (× 1.25).
Etched with saturated solution of ammonium persulfate.

instead of Brinell numbers because of the thinness of the slab used in making the tests. The results for the specimen from casting F5 showed that for two series of seven readings, each series taken at equal intervals along two lines midway between the center and the outside edge and parallel to one edge, the Rockwell hardness numbers, "B" scale, varied from 82 to 89 and from 80 to 85. These lines pass through the centers of adjacent test specimens. A series of 10 readings through the center of the specimen, but no readings within the center 0.5 in., gave a variation of 79 to 86. The maximum range in readings for either of the two traverses connecting the center lines of adjacent test specimens is 7 and since this is little more than twice the difference found when three readings were taken within about $\frac{1}{8}$ in. of each other the variation cannot be considered large.

TABLE I.—CHEMICAL COMPOSITIONS.

Each value is the average of duplicate analyses except as noted.

Element	Percentage of Element in Specimen	
	Specimen A1ZTGS	Specimen F1ZTGS
Silicon	1.75	1.65
Sulfur, evolved as H_2S	0.127	0.092
Sulfur, gravimetric[a]	0.138	0.099
Manganese	0.50	0.47
Phosphorus	0.48	0.47
Total carbon	3.32	3.20
Graphite[b]	2.56	2.40
Combined carbon (by difference)	0.76	0.80
Copper[b]	0.05	0.10
Chromium[b]	0.01	0.01
Nickel[b]	0.04	0.01
Molybdenum[b]	0.00	0.00

[a] Analysis from standard sample was 0.120 as compared with the certificate value of 0.122.

[b] Single analysis.

The chemical compositions of the irons used, requested by Mr. Dierker, are presented in the accompanying Table I. These analyses were made by the American Cast Iron Pipe Co. through the courtesy of Mr. MacKenzie. The material used in the analyses came from the ends of broken specimens adjacent to the mid-points of the lengths of the castings.

Mr. Lansford raised the question as to whether or not specimens having the same $\frac{t}{r}$ as the specimens tested but with larger values of t and r would give the same variation of modulus of rupture with $\frac{t}{r}$. The question cannot be answered because no tests of larger (or smaller) specimens have been made, though it is hoped that some tests of this character may be made at a later date. Messrs. Moore, Lansford, and Osgood all desire the substantiation of the dotted parts of the curves shown in Fig. 7. In the original planning of the tests it did not seem feasible to attempt to machine specimens having $\frac{t}{r}$ ratios less than 0.243. However some tests of other irons are contemplated which will include these smaller ratios, and it may also be possible to study elastic failure and buckling action in these future tests. The suggestion of Mr. Osgood to plot the $\frac{t}{r}$ values against some function of the modulus of rupture rather than against the modulus of rupture itself has been tried. The curves obtained were similar in form to those already shown and, at this stage of the study, did not seem to give any additional information.

THE EFFECT OF OVERSTRESSING AND UNDERSTRESSING IN FATIGUE

By J. B. Kommers[1]

Synopsis

This paper gives the damage curves obtained after the application of various numbers of cycles at 10, 20, and 30 per cent overstress. Results are given for three different carbon steels and one cast iron. The results include tests on standard smooth specimens and square-notched specimens.

The effects of overstress on the fatigue properties and the changes taking place in an ordinary fatigue test to failure are explained by means of the damage curves.

The effects of progressively increasing understress on the specimens which did not fail in the overstress tests are reported.

The effects of overstressing and understressing on the static properties of materials as determined subsequently to the fatigue tests are discussed.

In this discussion overstressing is used to mean stressing above the endurance limit, and understressing is stressing at or below the endurance limit.

In 1921, H. F. Moore and the author published[2] a few results on the effect of overstressing in fatigue of two different steels. In 1929 Smith, Connor, and Armstrong reported[3] some results on overstress which they stated contradicted the results obtained by H. F. Moore and the author. Since both sets of results were fragmentary, it seemed desirable to study the matter more thoroughly. It was expected also that the results would give fundamental information on the processes which are going on in any fatigue test. The tests were started in 1930 and have been carried on more or less continuously since then.

This investigation has been carried on as one of the research projects of the Department of Mechanics, College of Engineering, at the University of Wisconsin. The author is indebted to G. G. Thorp, of the Illinois Steel Co., of Chicago, Ill., and to J. T. MacKenzie, of the American Cast Iron Pipe Co., of Birmingham, Ala., for the materials used in the tests. The assistance of O. S. Peters, of Washington, D. C., for determining the tension test results on the cast iron, is gratefully acknowledged. Thanks are due the Wisconsin Alumni Research Foundation and the American Society for Testing Materials for part of the funds used in carrying on the investigation.

The first report of the present tests was published in *Engineering*.[4] That paper was submitted for publication in 1936, and since then additional results have become available on another carbon steel, on a cast iron, and especially on the effect of overstressing specimens having an imposed square notch.

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[1] Professor of Mechanics, University of Wisconsin, Madison, Wis.

[2] H. F. Moore and J. B. Kommers, "An Investigation of the Fatigue of Metals," *Bulletin No. 124*, University of Illinois (1921).

[3] J. H. Smith, C. A. Connor and F. H. Armstrong, "The Correlation of Fatigue and Overstress," *Journal*, Iron and Steel Inst. (British), Vol. 120, Part II, p. 267 (1929); *Engineering*, (London), Vol. 128, p. 605 (1929).

[4] J. B. Kommers, "Overstressing and Understressing in Fatigue," *Engineering* (London), Vol. 143, May 28 and June 11, 1937, pp. 620 and 676, respectively.

MATERIALS, TESTS, AND TEST SPECIMENS

The materials investigated were three different carbon steels and a cast iron. The chemical analysis of the materials is given in Table I, and some of the mechanical properties are given in Table II.

TABLE I.—CHEMICAL ANALYSIS.

Material	Carbon, per cent	Manganese, per cent	Silicon, per cent	Phosphorus, per cent	Sulfur, per cent
Steel Z	0.27	0.73	0.13	0.014	0.035
Steel Y	0.48	0.73	0.15	0.015	0.034
Steel X	0.62	0.36	0.18	0.020	0.040
Cast iron	3.57	0.58	1.58	0.66	0.06

TABLE II.—MECHANICAL PROPERTIES.

Material	Tensile Strength, lb. per sq. in.	Yield Point, lb. per sq. in.	Elongation in 2 in., per cent	Reduction of Area, per cent	Endurance Limit, Standard Specimen, lb. per sq. in.	Ratio, Endurance Limit, Standard Specimen, to Tensile Strength	Endurance Limit, Notched Specimen, lb. per sq. in.
Steel Z	78 500	52 200	34.3	62.1	34 000	0.43	21 600
Steel Y	123 900	66 700	17.0	30.4	51 200	0.41	24.600
Steel X	134 500	64 800	14.0	20.0	48 800	0.36	30 400
Cast iron	33 600	......	....	....	22 000	0.66	15 700

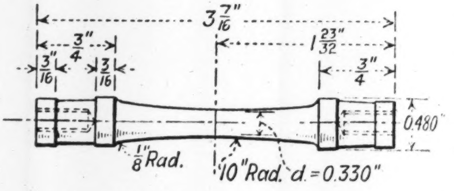
The carbon steel bars were received as $\frac{3}{4}$-in. round bars. The cast iron bars were cast in the form of $\frac{7}{8}$-in. round bars, about 15 in. long. All materials were tested as received.

The tension test specimens of the carbon steels were $\frac{1}{2}$ in. in diameter, and the cast iron tension specimens had diameters approximating the minimum diameter of the fatigue specimens, about 0.33 in.

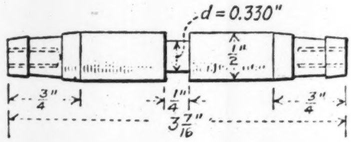
Steel Y showed some carbon segregation near the center of the bar, which was found to be several points higher than the average percentage for the whole cross-section. Rockwell hardness tests showed that the center of the bar had an average hardness of B71, while near the outer edge the hardness was B64.

Steel Z showed an average Rockwell hardness at the center of B51, and about B46 near the outer edge.

Steel X showed an average Rockwell hardness of about B67.



(a) Standard fatigue specimen.



(b) Notched fatigue specimen.

FIG. 1.—Fatigue Specimens.

The fatigue tests were carried out on R. R. Moore type machines, which subject rotating-beam specimens to completely reversed bending stress. The machines run at 1750 r.p.m., which means about 2,500,000 cycles in 24 hr. Figure 1 shows the standard smooth specimen and the square-notched specimen which were used in the tests. The standard specimens were turned, ground, and polished. The diameter of the notched specimen was made the same as the minimum diameter of the standard specimen for each material. The minimum diameters employed were 0.330 in. for the 0.27 per cent carbon steel and

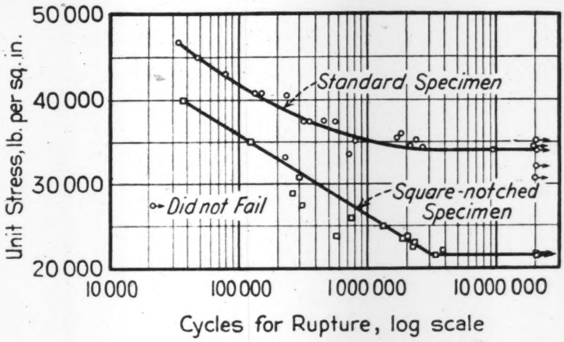


FIG. 2.—*S-N* Diagram for Steel Z.

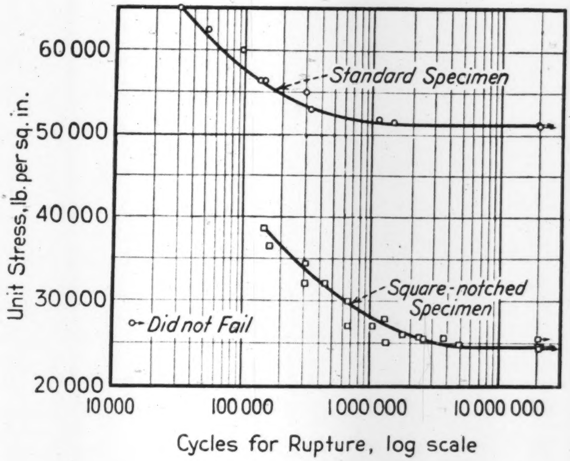


FIG. 3.—*S-N* Diagram for Steel Y.

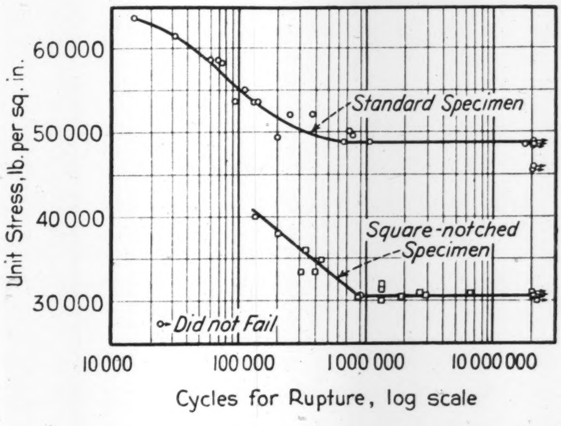


FIG. 4.—*S-N* Diagram for Steel X.

the cast iron, and 0.300 in. for the two higher carbon steels.

Figures 2, 3, 4 and 5 show the *S-N* diagrams for the four materials, for both standard and square-notch specimens. In these diagrams the unit stress, *S*, is plotted as ordinate to the natural scale, and the number of cycles required for failure, *N*, is plotted as abscissa to a logarithmic scale.

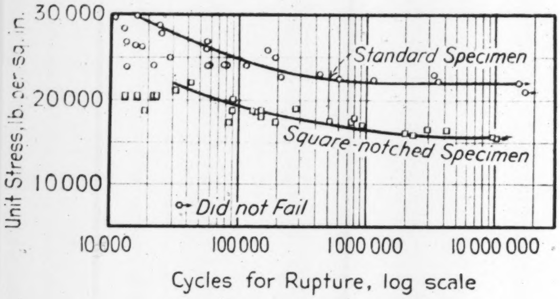


FIG. 5.—*S-N* Diagram for Cast Iron.

Figure 6 shows a conventionalized *S-N* diagram. The processes which cause final failure of a material are evidently taking place at the stresses and numbers of cycles corresponding to the shaded portion of this diagram. Information is very much needed as to the nature of the processes which cause final failure and as to the changes which these processes cause in the properties of a material.

In Fig. 6 suppose that the line *AB* represents an overstress of 10 per cent above the original endurance limit. The point *B* represents the number of cycles at which the specimen fails. Such points as *B*, for 10, 20, 30, etc., per cent of overstress may be obtained from Figs. 2 to 5. When the specimen is subjected to a period of overstress up to any point *C*, the ratio that this number of cycles bears to the number corresponding to *B* will be called the "cycle ratio," and will be expressed in per cent. The number of cycles corresponding to point *B* will therefore be called a cycle ratio of 100 per cent, and the number corresponding to any point *C* may be anything less than 100 per cent. So far as the author is aware, this term "cycle ratio" has not been employed hitherto in fatigue testing, but it has been found very useful in plotting the results of overstress tests.

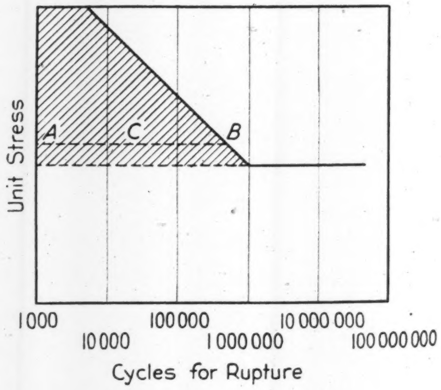


FIG. 6.—Conventional *S-N* Diagram.

In the present tests a series of specimens was subjected to a definite cycle ratio at a definite percentage of overstress. When the specimen had been subjected to the number of cycles corresponding to the cycle ratio, the load on the specimen was reduced in order to determine the value of the new endurance limit. Enough specimens were tested to determine the new endurance to the nearest 1 per cent of the virgin endurance limit. Specimens were tested both above and below the new endurance

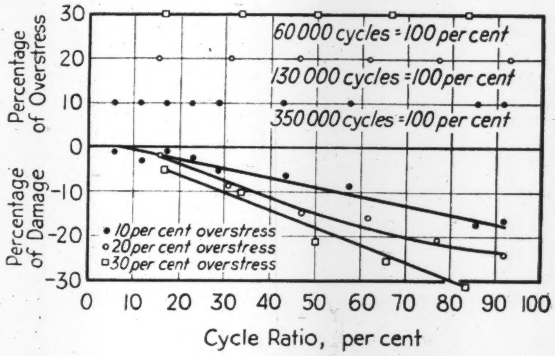


FIG. 7.—Steel Z, Standard Specimen.

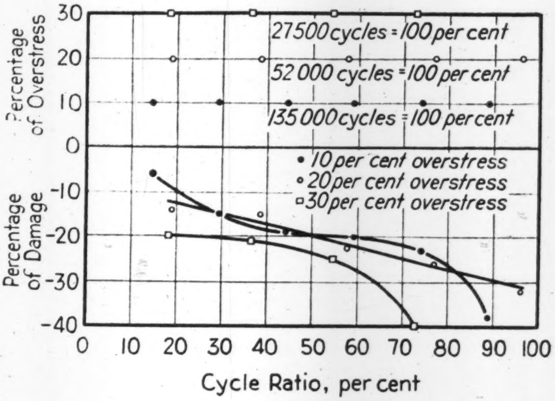


FIG. 8.—Steel Y, Standard Specimen.

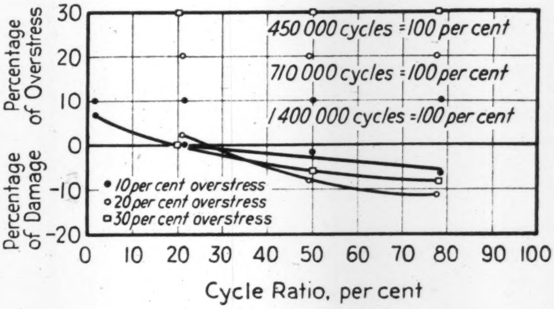


FIG. 9.—Steel Y, Square-Notched Specimen.

limit. The amount by which the new endurance limit was below the virgin endurance limit is called the percentage of damage. By choosing a series of cycle ratios, data were obtained for plotting a damage curve at a definite percentage of overstress. In order to determine the trend of a damage curve for a given material, it was found desirable to choose cycle ratios which approached quite

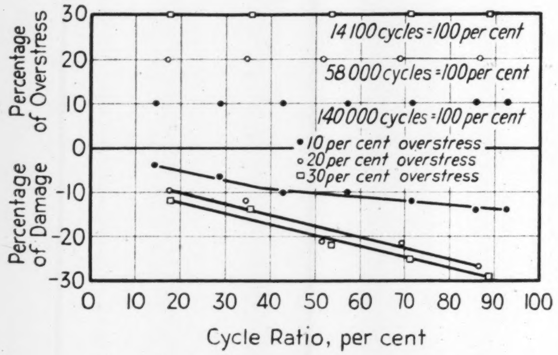


FIG. 10.—Steel X, Standard Specimen.

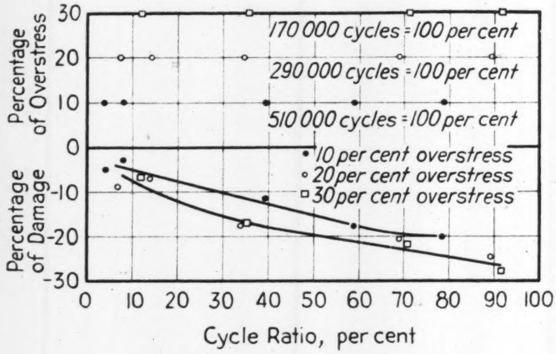


FIG. 11.—Steel X, Square-Notched Specimen.

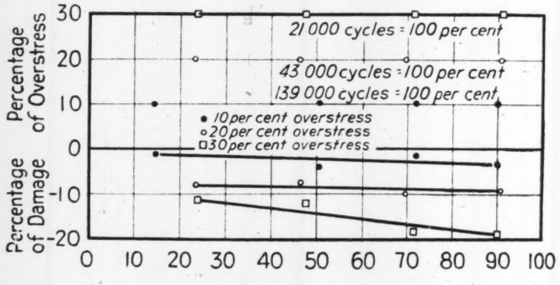


FIG. 12.—Cast Iron, Standard Specimen.

closely points such as *B* in Fig. 6, because small differences in number of cycles sometimes produced results which were not quite consistent. In this manner damage curves were obtained for 10, 20, and 30 per cent overstress.

Tests such as outlined above, in effect, break up the fatigue test into stages. By determining the new endurance limit at the different cycle ratios it is possible to determine what the fatigue process is doing in changing the fatigue properties of a material on its journey toward failure.

If tests of this kind were carried out on a direct-stress fatigue testing machine, it would be possible to determine also the new static properties after a material has been subjected to various amounts of overstress at different cycle ratios. The new static properties cannot be determined after a rotating-beam fatigue test because in this test the cross-section of the specimen is not subjected to uniformly distributed stress.

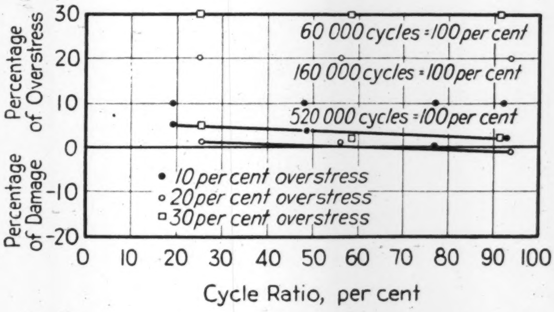


FIG. 13.—Cast Iron, Square-Notched Specimen.

The method that French[5] has suggested for determining the "probable damage curve" for a material, determines the overstress and the corresponding number of cycles which will not reduce the endurance limit below its original value. The present tests go further than this, because they determine not only whether the overstress causes damage, but, by determining the new endurance limit, they disclose exactly how much damage has been done at a given overstress and cycle ratio.

TEST DATA AND RESULTS

Figures 7 to 13 show the damage curves obtained for the materials tested, at 10, 20, and 30 per cent overstress. For each material, except the 0.27 per cent carbon steel, steel Z, curves are shown for the standard specimen and the square-notched specimen. In these figures the abscissa is the cycle ratio, and above the horizontal zero line is plotted the percentage of overstress. Below the zero line is plotted the percentage of damage, the lower points being vertically below the upper points. In each figure the number of cycles corresponding to a cycle ratio of 100 per cent is shown for 10, 20, and 30 per cent overstress.

It will be noted that quite a few of the damage curves tend to approach a horizontal asymptote at high cycle ratios. In other cases the damage curves are practically straight lines, and in Fig. 8 two of the curves definitely turn downward at the higher values of cycle ratio.

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[5] H. J. French, "Fatigue and the Hardening of Steels," *Transactions*, Am. Soc. Steel Treating, Vol. 21, p. 899 (1933).

It is evident that in a number of cases an increase in percentage of overstress does not show a proportionate increase in the amount of damage. For instance, in Fig. 10 the damage curves for 20 and 30 per cent overstress are close together. In Fig. 11 a single curve has been drawn through the points for 20 and 30 per cent overstress. In Fig. 13, a single curve has been used for 10 and 30 per cent overstress, and this is close to the curve for 20 per cent overstress. Figure 13 also shows the curious case of the 30 per cent overstress curve lying higher than that for 20 per cent overstress.

In Figs. 8 to 13 it is evident that the percentage of damage for the standard specimens is greater than that for square-notched specimens. This was true also for steel Z, but there were not enough results available to outline properly the curves for this steel.

Some of the curves cross the axis for zero damage, and some of the others, if extended, would cross the zero axis. This crossing point indicates the cycle ratio up to which the overstress causes no damage, and within which the overstress may actually increase the endurance limit.

The curves for the cast iron with square-notched specimens are unique in the fact that all the points except one lie on or above the line for zero damage. This indicates that for this particular cast iron, square-notched specimens are not damaged by overstress, even when the cycle ratio is over 90 per cent. This implies an unusual ability to withstand overstress.

Table III has been prepared for a convenient comparison of damage results, showing the approximate percentages of damage for 10 per cent overstress at cycle ratios of 20, 50, and 80 per cent, for both standard and square-notched specimens, and, below, similar results for 20 and 30 per cent overstress, respectively.

In Fig. 9 the curve for 10 per cent overstress extends back almost to zero cycle ratio, and will be discussed in greater detail. The original endurance limit of this square-notched steel was 24,600 lb. per sq. in. The curve shows that at 10 per cent overstress and 1.4 per cent cycle ratio, which in this case meant 20,000 cycles, the effect of the overstress was to increase the virgin endurance limit 7 per cent. Continuing the test to a cycle ratio of about 21 per cent, corresponding to 300,000 cycles, reduced the endurance limit to its original value. At a cycle ratio of 50 per cent, corresponding to 700,000 cycles, the original endurance limit had been reduced by 3 per cent. At a cycle ratio of 80 per cent, corresponding to about 1,120,000 cycles, the original endurance limit had been reduced by about 6 per cent.

TABLE III.—DAMAGE DUE TO OVERSTRESS.

Plus signs indicate endurance limit increased, minus signs indicate endurance limit decreased.

Material	Standard Specimen			Square-Notched Specimen		
	20 per cent Cycle Ratio	50 per cent Cycle Ratio	80 per cent Cycle Ratio	20 per cent Cycle Ratio	50 per cent Cycle Ratio	80 per cent Cycle Ratio
10 PER CENT OVERSTRESS						
Steel Z	−3	−9	−15	....	....	....
Steel Y	−10	−20	−26	0	−3	−6
Steel X	−5	−10	−13	−8	−15	−20
Cast iron	−1	−2	−3	+5	+4	+3
20 PER CENT OVERSTRESS						
Steel Z	−4	−15	−22	....	....	....
Steel Y	−13	−20	−28	+3	−8	−11
Steel X	−11	−18	−25	−12	−19	−24
Cast iron	−8	−8	−9	+1	0	−1
30 PER CENT OVERSTRESS						
Steel Z	−7	−18	−30	....	....	....
Steel Y	−20	−24	>−45	0	−6	−8
Steel X	−13	−20	−27	−12	−20	−24
Cast iron	−11	−14	−18	+5	+4	+3

These results in Fig. 9 indicate, therefore, that for cycle ratios up to 21 per cent, the overstress may actually increase the endurance limit. However, as the test to failure proceeds, the fatigue properties are gradually changed, the endurance limit being decreased as indicated.

In this same Fig. 9, at a cycle ratio of 80 per cent, the endurance limit has been reduced 6, 11, and 8 per cent, respectively. At this point in the test, therefore, 10, 20, and 30 per cent overstress above the virgin endurance limit, represent true overstresses above the then existing endurance limits of about 11, 22, and 33 per cent. There is a cumulative effect here, which, near the end of the test, would tend to make the progress toward failure very rapid indeed. If a crack is developed near the end of the test, there are additional stress concentration effects, which would further tend to accelerate progress toward failure.

The question may be asked why it is that the damage curves for the notched specimens for each material show smaller percentages of damage than the curves for the standard specimens at the same cycle ratio. This is probably due to the fact that the nominal unit stress calculated on the basis of the diameter of the notched specimen is not the true stress which exists at the sharp corner where failure occurs. The endurance limit for the standard specimen of cast iron was 22,000 lb. per sq. in., whereas it was 15,700 lb. per sq. in for the notched specimen, which indicates a stress-concentration factor of 1.4.

If it is now assumed that for all nominal stresses applied to the notched specimen above the endurance limit the true stress may be found by multiplying by 1.4, it is possible to plot an *S-N* curve for these true stresses and the number of cycles which caused failure in the notched specimens. It was found that this *S-N* curve lies to the right of the *S-N* curve for standard specimens, and the two curves merge only at the endurance limit.

It is obvious then that the application of 100,000 cycles of 10 per cent overstress means a considerably larger cycle ratio for the standard specimen than for the notched specimen, since the point of failure on which cycle ratio is calculated lies further to the right for the notched specimen than for the standard specimen.

It may be that if the stress-concentration factor is 1.4 at the endurance limit, this factor is progressively less than 1.4 as the stresses are increased above the endurance limit.

True stresses similar to those mentioned above were calculated also for the Steel X, for which the stress-concentration factor was about 1.6. The *S-N* curve for these true stresses was again found to lie to the right of the curve for standard specimens, and the discrepancy was much greater than it was for cast iron. Since all the stresses employed in this case were less than the yield point of the material, it might be assumed that the stress-concentration factor would be more likely to be constant for all stresses. Evidently this was not the case.

The value of damage curves lies in their approximate position, their trend, and the evidence they present of what is going on in any fatigue test to failure. Too much stress should not be laid on an individual result or even on an individual curve. Each curve is based upon cycle ratios calculated on the basis of points such as *B* in Fig. 6, which are obtained from the *S-N* diagrams. Some judgment must be used in drawing an average *S-N* curve through the plotted points, and this curve determines where the point *B* lies at a given overstress.

Another consideration to be borne in

mind is the fact that it is precisely in number of cycles that there may be a considerable difference in fatigue tests even at the same unit stress. For instance, five specimens of steel Z were all tested to failure at a unit stress of 37,400 lb. per sq. in. The specimen showing the least endurance failed at 315,000 cycles, while the strongest failed at 553,000 cycles, a difference of over 230,000 cycles. These differences may be due to lack of homogeneity between specimens, and perhaps to slight differences of surface finish. Such effects would, of course, influence the results obtained from overstress tests.

UNDERSTRESSING RESULTS

For specimens which did not fail in the overstressing tests, the maximum number of cycles employed was 20,000,000 at first, but this was reduced to 10,000,000 in the later tests. Since the *S-N* curves in Figs. 2 to 5 become horizontal at less than 10,000,000 cycles, evidently this number is sufficient to determine the endurance limit. However, for some of the specimens subjected to large cycle ratios, the test was continued to 20,000,000 cycles, to make certain that failure would not occur.

All specimens which did not fail were subjected to further test, which usually consisted in adding a load of 1 lb. per day. The specimen was thus subjected to progressively increasing understress, each period consisting of approximately 2,500,000 cycles. These tests showed that the damage that had been done by overstress could be repaired by progressively increasing understress. Sometimes the endurance limit which had been decreased by overstress was restored to its original value, sometimes it was increased to the value of the original overstress, and sometimes it was increased above the original overstress by substantial percentages.

These tests show that the damage done by overstress is not always permanent. The tests also show that the endurance limit of a material is not a fixed quantity. The endurance limit can be manipulated by various stressing operations. For the smaller cycle ratios and the smaller percentages of overstress, the endurance limit can be increased by overstress; for the larger cycle ratios and percentages of overstress it is usually decreased by overstress; and it can be increased especially by progressively increasing understress.

The following summary shows some of the highest values of strengthening obtained after overstress followed by progressively increasing understress. The results are based on a maximum run of 2,500,000 cycles at any one unit stress.

Material	Range of Overstress, per cent	Range of Cycle Ratio, per cent	Range of Strengthening, per cent
Steel Z:			
Standard specimen.......	10	6 to 96	7 to 16
Square-notched specimen..	10 to 30	4 to 55	15 to 45
Steel Y:			
Standard specimen........	10	7 to 74	9 to 20
Square-notched specimen..	10 to 30	1 to 79	15 to 39
Steel X:			
Standard specimen........	10 to 20	7 to 71	11 to 25
Square-notched specimen..	10 to 20	8 to 14	12 to 22
Cast Iron:			
Standard specimen........	10 to 30	23 to 72	10 to 16
Square-notched specimen..	10 to 30	19 to 94	11 to 17

OTHER RESULTS ON THE EFFECT OF OVERSTRESS AND UNDERSTRESS

The tests on cyclic overstressing and understressing which have been previously discussed have made plain the effects of such stressing on the subsequent endurance properties. Another way in which valuable information may be obtained is to determine the static properties of materials after they have been subjected to overstress and understress.

Mehmel's[6] tests on concrete are interesting in this connection. Specimens were subjected to compression from a minimum to a maximum stress in fatigue. When the maximum stress was below the endurance limit, both the elastic deformation and the permanent set finally became constant. In a subsequent static test the elastic behavior was studied. When the specimens were subjected to repeated stresses above the endurance limit, the deformations did not become stable.

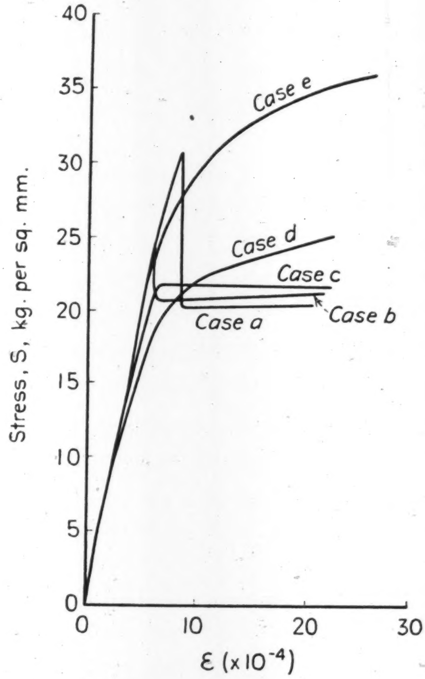

FIG. 14.—Results on Annealed 0.05 per cent Carbon Steel (Memmler and Laute).

Mehmel concluded that when stability was attained by the deformations, and when the ultimate static strength had not been reduced by the repeated stresses, then the applied stress was less than the endurance limit. Under these conditions a linear relation existed between unit stress and unit deformation up to the applied maximum stress and even higher. On the other hand, when the maximum stress was above the endurance limit stable conditions of deformation were not attained, and the stress-deformation curve became concave upward.

TABLE IV.—ANNEALED 0.05 PER CENT CARBON STEEL.

	Overstressing and Understressing		Yield Point at 0.2 per cent Set, kg. per sq. mm.	Upper Yield Point, kg. per sq. mm.	Lower Yield Point, kg. per sq. mm.	Elastic Limit at 0.003 per cent Set, kg. per sq. mm.	Tensile Strength, kg. per sq. mm.	Elongation, per cent	Reduction of Area, per cent
	$\epsilon \times 10^{-4}$	N, millions							
Case *a*	0	0	20.4	30.7	20.4	24	37.4	35.5	78.5
Case *b*	8	39.5	21.0	24.2	20.6	24.2	37.7	33.0	77.7
Case *c*	12	40.5	21.7	21.7	21.7	17.3	37.7	33.0	77.7
Case *d*	14.4	0.03	24	....	....	9.7	37.7	19.0	76
Case *e*	16.0	0.03	34.2	....	....	22.5	40.4	13.0	76.5

Memmler and Laute[7] also studied the effect of cyclic overstress and understress by means of static tests made subsequent to the fatigue tests. Thus they determined the changes in static properties which the repeated stresses had caused. The results obtained on an annealed 0.05 per cent carbon steel are shown in Fig. 14 and Table IV.

Memmler and Laute employed a constant strain fatigue machine, and the endurance limit of this material for completely reversed direct stresses in tension and compression corresponded to a unit deformation, ϵ, of 12.8×10^{-4}. Case *a* in Fig. 14 shows the static properties of a specimen which was neither overstressed nor understressed. In case *b* the specimen was first subjected to 39,500,000 cycles of stress less than the endurance limit. The static results show that this understress had its most significant effect on the upper yield point, which was reduced 21 per cent. Neither the tensile strength nor the ductility was affected in a significant manner. In case *c* the unit stress was only slightly below the endurance limit and was applied for 40,500,000 cycles. This action reduced the upper yield point 29 per cent, in such manner than the upper and lower yield points were made equal. The elastic limit at 0.003 per cent permanent set was also reduced by a large percentage, but the tensile strength and the ductility were not much affected.

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[6] A. Mehmel, "Untersuchungen über den Einflusz häufig wiederholter Druckbeanspruchungen auf Druckelastizität und Druckfestigkeit von Beton," *Mitteilungen* aus dem Institut für Beton und Eisenbeton an der Technischen Hochschule, Karlsruhe (1926).

[7] K. Memmler and K. Laute, "Dauerversuche an der Hochfrequenz-Zug-Druck-Machine (Bauart-Schenck)," *Forschungsarbeiten* auf dem Gebiete des Ingenieurwesens, No. 329 (1930).

This is a very clear and convincing demonstration of the manner in which repeated stresses produce the changes by means of which the "natural elastic limit" of Bauschinger is established. This "natural elastic limit" or endurance limit corresponded to a deformation of 12.8×10^{-4}. The repeated stresses at a deformation of 12×10^{-4} reduced the elastic limit at 0.003 per cent set from 24 to 17.3 kg. per sq. mm. This result may be contrasted with that obtained by Mehmel on concrete, in which cyclic understress caused the material to become elastic up to and somewhat above the stress applied. In the case of steel, therefore, cyclic understress decreased the elastic limit, and in the case of concrete understress increased the elastic limit.

It is evident from the above tests on steel that the elastic limit and yield point properties are the ones most influenced by cyclic understress, and the results give further reasons why there is little correlation between these static properties and the endurance limit of a material. The tensile strength and the ductility are influenced to a much smaller degree than are the elastic limit and yield point.

In case *d* in Fig. 14 the specimen was subjected to a unit stress above the endurance limit. This overstress of 12.5 per cent was applied for 30,000 cycles, after which the static properties were determined. The yield point at 0.2 per cent permanent set had been increased 17.5 per cent, and the elastic limit at 0.003 per cent set was reduced 60 per cent. The tensile strength was only slightly changed, but the ductility as represented by percentage of elongation was reduced 46 per cent.

In case *e* a still higher unit stress, amounting to 25 per cent overstress, was applied for 30,000 cycles. The static test showed that this overstress had greatly increased the yield point at 0.2 per cent set, and that the elastic limit at 0.003 per cent set was slightly decreased. The tensile strength was somewhat increased, but the ductility represented by percentage of elongation was decreased 63 per cent.

Further tests may show that the drastic reduction of percentage of elongation by cyclic overstress is a significant factor in the reduction of endurance limit which overstress usually produces. It may be that this lack of ductility prevents a material from adjusting itself to a subsequent repeated stress unless that stress is quite small.

Memmler and Laute made similar tests on annealed nickel. They found that understressing raised the S-ϵ curves in the neighborhood of the yield point. This increase was greater for 80,400,000 cycles than for 1,000,000 cycles. This effect on the S-ϵ curves, however, decreased as the ultimate strength was approached in the static tests, since the ultimate remained practically unchanged.

Understress tests on copper gave results similar to those reported by Mehmel for concrete, since the elastic limit was increased. Tensile strength was increased slightly and ductility was decreased slightly. Overstress had the effect of increasing the elastic limit considerably, and the yield point and ultimate strength slightly.

The effects of completely reversed cyclic stresses at or just below the endurance limit may be summarized as follows: The greatest effect of understress on the static properties is produced at the elastic limit and yield point. When the material has an upper and lower yield point the effect of understress is to reduce the upper yield point until finally the upper and lower yield point are equal. The final yield point may be slightly higher than the original lower yield point. Understress may increase the tensile strength somewhat and decrease the ductility as represented by percentage of elongation and reduction of area.

Understress will usually increase the endurance limit, and Memmler and Laute found that the number of cycles necessary must exceed the number at which the S-N curve becomes horizontal. However, even with increase of cycles, the amount of strengthening finally reaches a maximum value. This was clearly shown by the author[8] for a gray cast iron with a tensile strength of 20,000 lb. per sq. in. The percentage of strengthening obtained from a constant unit stress just below the endurance limit increased with the number of cycles up to 15,000,000. Beyond this there was no further strengthening, even though the number of cycles was as high as 40,000,000. The optimum number of cycles for a maximum increase of endurance limit at a constant unit stress varies with different materials.

When a constant unit stress has increased the endurance limit, it is possible to obtain further strengthening by increasing the applied unit stress. With such progressively increasing understress it is possible to obtain very large percentages of increase above the virgin endurance limit. For increasing the endurance limit by understress, the most effective stress is one at or just below the original endurance limit. This was clearly shown by the results which the author[8] obtained on gray cast iron.

In 1910 Bairstow[9] reported some results on axle steel subjected to repeated axial tensile stresses from zero to a maximum value. He showed how the repeated stresses changed the shape of the S-ϵ curve in a subsequent static test.

At the National Physical Laboratory in England, H. J. Gough[10] and his colleagues used a quite different method from that of the author in investigating the changes which take place under repeated stresses. Precise X-ray diffraction methods were used to determine the changes produced in the crystalline structure of metals by plastic deformation and fracture.

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[8] J. B. Kommers, "The Effect of Under-Stressing Cast Iron and Open-Hearth Iron," *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part II, p. 368 (1930).

[9] L. Bairstow, "The Elastic Limits of Iron and Steel Under Cyclical Variations of Stress," *Transactions*, Royal Soc. (London), Vol. 210A, p. 35 (1910).

[10] H. J. Gough and W. A. Wood, "A New Attack upon the Problem of Fatigue of Metals, Using X-ray Methods of Precision," *Proceedings*, Royal Soc. (London), Vol. 154A, p. 510 (1936).

H. J. Gough, "Characteristics of the Deformation and Fracture of Metals as Revealed by X-rays," *The Engineer*, April 20, 1937, p. 520.

CONCLUSIONS

1. The endurance limit of a material is not a fixed quantity. It may be manipulated by various stressing methods. Periods of cyclic overstress quite commonly reduce the endurance limit, while periods of progressively increasing cyclic understress may greatly increase the endurance limit.

2. Overstress may either increase or decrease the endurance limit. Usually the endurance limit is increased only at the smaller values of cycle ratio and overstress. However, one material, square-notched cast iron, showed practically no damage due to overstress up to 30 per cent. Cycle ratio is defined as the ratio of the number of cycles applied at a given overstress to the number of cycles at which the material would fail at that stress.

3. The two important factors which determine the amount of damage due to overstress seem to be the percentage of overstress and the cycle ratio:

4. In some cases the damage curves tend to approach a horizontal asymptote at high cycle ratios, in other cases the damage curves are practically inclined straight lines, and in still other cases the damage increases rapidly at high cycle ratios.

5. Progressively increasing cyclic understress, applied to a specimen after overstress, not only may repair the damage which has been done, but in some cases may augment the original endurance limit by substantial percentages. In some cases the endurance limit may be raised to a value considerably above that of the original overstress. A material may be strengthened even after large cycle ratios of overstress.

DISCUSSION

MR. J. T. MACKENZIE.[1]—I think these are the first fatigue tests where the same size specimen was used both in the tension and in the fatigue test. In most of them the 8-in. tension test specimen has been used against the 0.33-in. fatigue specimen. I think that is probably the explanation for the rather high ratio of fatigue to tensile strength as compared to those previously obtained. There is one point, however, in that ratio which seems to me to carry some danger, and that is, the modulus of rupture of that same cast iron in flexure would be probably twice the tensile strength, which would reduce the ratio to 0.33. Now Mr. Thum made a study of the distribution of stress in the flexure test, with special reference to the fatigue test, and determined that there probably would be only about 14 per cent excess calculated stress at the stresses employed in the fatigue test, that is, at the fatigue limit, so that possibly the values would be only 14 per cent too high. The results on the notched cast-iron specimens bear out what we know about the flexure test. In some results that you will find in the Collins and Draffin paper, the metal close to the highest stressed portion of cast iron apparently does have quite an effect in reinforcing those highly stressed parts. That is, of course, the explanation for the high modulus of rupture in flexure, and as this paper shows, the high value of the ratio of notched to unnotched specimen.

MR. H. F. MOORE.[2]—The fatigue limit, to my mind, is a function of the stress history of the material—among other things—and, of course, the precise stress history of the material in structural and machine parts is very variable and quite unpredictable in most cases. I believe that the usefulness of the fatigue limit to designers and users of metal parts may be justified by what may be characterized as bad scientific reasoning, but good engineering reasoning. Mr. Kommers and others have shown that long periods of repeated stress below the fatigue limit *increase* the fatigue limit beyond that of the virgin metal. Now long periods of understress usually occur in structural and machine parts. Therefore, although the fatigue limit is not a scientific property of the metal which "stays put" throughout the period of service of a structural or machine part, it seems to be probably a more severe criterion of the strength of the metal than the fatigue limit at any given instant during that period—unless the metal has been subjected to a rather large number of serious overstresses. It seems to be a reasonably safe guide for the designer, if the effect of "stress raisers" are taken into account.

It may be added that, to a less degree, all our common mechanical properties of metals—yield strength, tensile strength, energy for fracture—might well be subjected to a similar question.

I am sorry that I have not at hand the data to answer Mr. MacKenzie's question concerning the modulus of rupture of the cast iron Mr. Kommers tested. I do know that when I have run flexure tests of round cast-iron bars I have found

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[1] Chief Chemist, American Cast Iron Pipe Co., Birmingham, Ala.

[2] Professor of Engineering Materials, University of Illinois, Urbana, Ill.

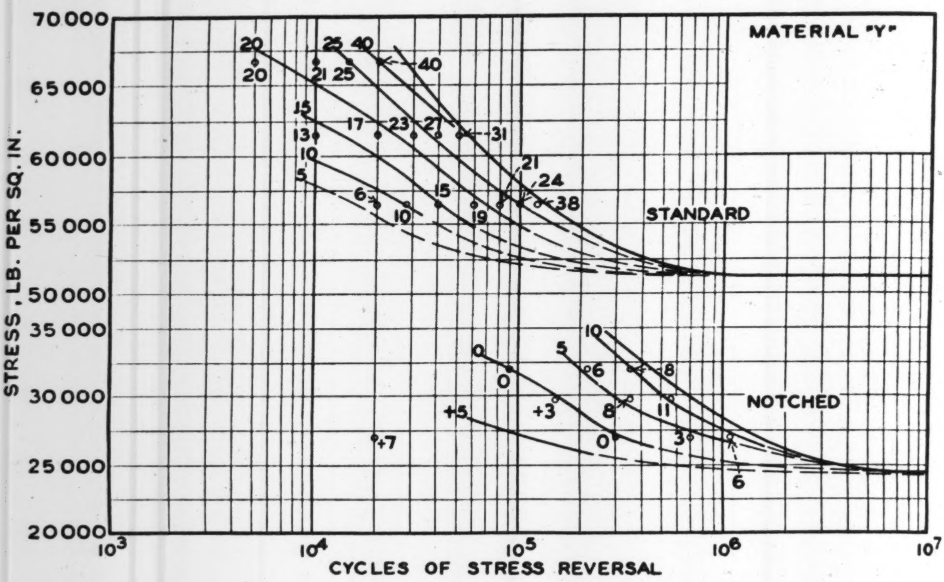

FIG. 1.—Overstress Data for Steel Y Plotted to Conventional Semilogarithmic Coordinates.

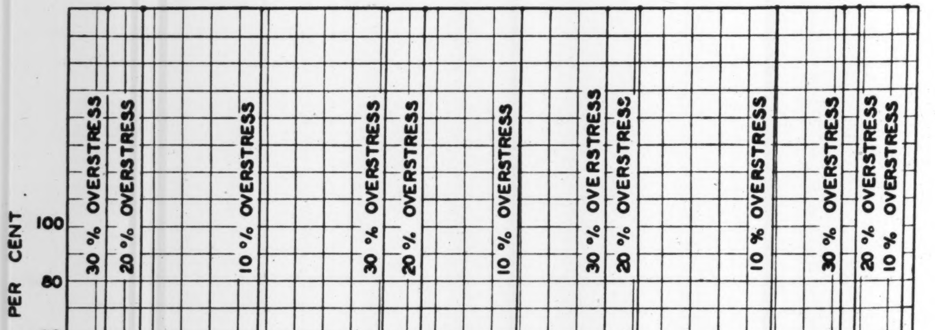

FIG. 2.—Overstress Data for Steel Y and Cast Iron Plotted as Percentage of Damage *versus* Cycles of Overstress for 10, 20 and 30 per cent Overstress.

that commonly the modulus of rupture exceeds the tensile strength by more than 10 per cent—sometimes by 50 per cent. I am inclined to think that with such an inhomogeneous and imperfectly elastic material as gray cast iron it is to be expected that when formulas based on the assumptions of the theory of elasticity are used, discrepancies between computed and test values may be expected to be large.

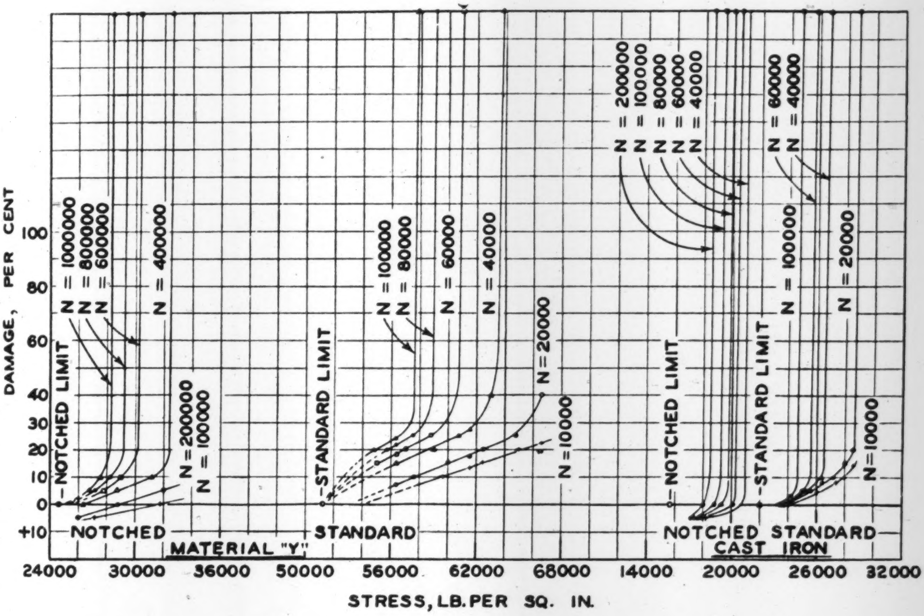

FIG. 3.—Overstress Data for Steel Y and Cast Iron Plotted as Percentage of Damage *versus* Stress for Various Constant Cycles of Overstressing

MR. A. L. SANFORD[3] (*by letter*).—The overstressing results presented in this paper are, it is believed, the first attempt systematically to break down the area to the left of the upper portion of the *S-N* curve to see when damage due to overstressing starts and how that damage progresses with increased amounts of overstress or increased cycles of overstressing. Previous authors[4–8] have more or less limited their work to establishing the probable "damage line" of French, which corresponds to 0 per cent damage in Mr. Kommer's work, and have not attempted to study the area enclosed by the French "damage line" and the *S-N* curve. After reading this paper and realizing the enormous amount of work it must have entailed, Mr. Kommers is certainly to be congratulated for really pioneering the field of damage due to overstressing in the fatigue of metals.

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[3] Metallurgist, Battelle Memorial Institute, Columbus, Ohio.

[4] H. W. Russell and W. A. Welcker, Jr., "Damage and Overstress in the Fatigue of Ferrous Materials," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 118 (1936).

[5] H. J. French, "Fatigue and the Hardening of Metals," *Transactions*, Am. Soc. Steel Treating, Vol. 21, p. 899 (1933).

[6] H. F. Moore and J. B. Kommers, "An Investigation of the Fatigue of Metals," *Bulletin 124*, University of Illinois (1921).

[7] J. H. Smith, C. A. Connor and F. H. Armstrong, "The Correlation of Fatigue and Overstress," *Journal*, Iron and Steel Inst., Vol. 120, Part II, p. 267 (1929).

[8] H. B. Wishart and S. W. Lyon, "Effect of Overload on the Fatigue Properties of Several Steels at Various Low Temperatures," *Transactions*, Am. Soc. Metals, Vol. 25, p. 690 (1937).

While studying the paper, it was found that if the data for the four materials were replotted in various ways, some interesting relationships were encountered. Only the replotted data for steel Y and portions of the data for the cast iron are reproduced in the accompanying Figs. 1 to 3. The other materials showed the same relationships.

In Fig. 1 the data have been plotted in the conventional semilogarithmic manner with per cent damage lines drawn through points of equal damage due to overstressing. The damage lines assumed the proportions of a family of curves in each case, and in general the distance between lines of successively higher equal increments of damage became less the closer the *S-N* curve was approached. This seems rational since any point on the *S-N* curve above the endurance limit represents infinitely great damage or failure at the particular value of overstress in question.

The damage lines in the accompanying Fig. 1 in the region from 0 to 10 per cent overstress have been drawn (dashed sections) so that the lines gradually approach each other as the amount of overstress is decreased and ultimately come together at the endurance limit (note the exception of the + 5 per cent line for the square-notched condition). Perhaps only the 0 per cent damage line should approach the endurance limit and the others come into the *S-N* curve at some slightly greater stress, since no damage occurs at the endurance limit by definition. Also, strengthening may occur at the endurance limit to the extent that after running, say 10^7 cycles, a specimen is then capable of running much longer at some higher stress than a virgin specimen. Also, as the author points out, some judgment must be used in drawing the *S-N* curve through the plotted points, so that perhaps too much discussion of damage due to overstressing at very high cycle ratios is not in order, since large discrepancies may occur.

The + 5 per cent damage line for the square-notched specimens of steel Y represents overstressing which caused no damage but rather an increase in the endurance limit. That increased endurance limits can be obtained by understressing is well known. Is it not possible that overstressing which increases the endurance limit and understressing are the same? Assuming this, the + 5 per cent line has been drawn to some stress slightly below the endurance limit rather than meeting the other lines at the endurance limit.

In general, replotting the data in the manner of Fig. 1 enabled the writer to visualize more clearly the relationship between the *S-N* curve and the damage due to cyclic overstressing at 10, 20, and 30 per cent of overstress for various cycle ratios.

As pointed out by Russell and Welcker[4] two processes, namely, a strengthening process, and a damaging process, are at work in fatigue. The former is probably some manner of strain hardening, while the nature of the latter is unknown, but is probably of two stages, crack formation and crack propagation, the latter of which is very rapid in the final stages of fatigue. Since Mr. Kommers' data are the first to go beyond 0 per cent damage, the data were replotted in the forms shown in Figs. 2 and 3 to see whether any information regarding the rate of crack propagation would result. The data for the cast iron have been included to show the type of curves obtained for a material which is more resistant to overstressing, particularly in the notched condition, than most ferrous materials.

If we assume, which may be assuming too much, that the slope of the curves in Figs. 2 and 3 at any point is related to the rate of crack propagation, some interesting relationships are obtained. In general, increased overstress results in greater slopes in the lower portions of the curves in Fig. 2, and increasing the number of cycles of overstress in Fig. 3 results in the same increased slope. All of the curves show sharp upward breaks near the points of 100 per cent cycle ratio or infinite damage and failure. These breaks are also sharper with increased values of overstress in Fig. 2 or increased cycles of overstress in Fig. 3 as might be expected if the slopes are related to the rates of notch propagation.

The curves for the square-notched cast iron are particularly interesting, since the slopes from zero to infinite damage are almost infinite, indicating extremely rapid notch propagation once the inherent "stress raisers," which make the cast iron relatively insensitive to notches, are overcome.

MR. E. H. SCHULZ[9] (*as translated and abstracted by the author*).—Attention is called to a paper[10] by Müller-Stock, in which the effect of overstressing was studied by means of a different method. A specimen was first subjected to an overstress for a given number of cycles. The percentage of damage due to this overstress was obtained by subjecting the specimen to a still higher stress, and comparing the decrease in number of cycles for failure at this higher stress with the number of cycles required for failure of a virgin specimen at the same stress. Mr. Schulz points out a number of results which were similar to those obtained by the author.

MR. J. B. KOMMERS[11] (*author's closure by letter*).—Mr. MacKenzie refers to the fact that for the standard specimens of cast iron the ratio of endurance limit to tensile strength was high. There is another reason for this in addition to the one he gives. Figure 5 shows that the cast iron gave some erratic results. I have made some further tests on this cast iron in the region of the endurance limit. These test results were also erratic and indicated that, for some of the specimens at least, the endurance limit is 21,000 lb. per sq. in. or even less, instead of 22,000 lb. per sq. in. as given in Table II.

The A.S.T.M. Report on Impact Testing of Cast Iron, published in 1933,[12] shows that the average ratio of endurance limit to tensile strength was about 0.50, and the average ratio of endurance limit to modulus of rupture was about 0.23.

I wish to thank Mr. Sanford for the contribution of his Figs. 1 to 3, which enable further relationships to be visualized. In connection with his Figs. 2 and 3, he makes a tentative suggestion with respect to rate of crack propagation. I do not know whether the damage which occurs with increase of cycles of overstress may be called crack propagation. I say this because of the fact that with progressively increasing understress I succeeded in numerous cases in repairing the damage done by overstress, so that the endurance limit was restored to its original value or even higher. The knowledge which we have at present of what the fatigue process does is still incomplete, and the purpose of this paper was to add somewhat to our information.

Some further tests which have been carried out may be of interest in connection with Mr. Sanford's discussion. Square-notched specimens of ingot iron were subjected to 10 per cent overstress for various cycle ratios. At a cycle ratio of 10 per cent the damage was about 5 per cent, but the damage then decreased, so that at a cycle ratio of 55 per cent, the new endurance limit was about 1 per cent higher than that of a virgin specimen. This indicated more damage for the smaller than for the greater number of cycles. For cycle ratios greater than 55 per cent, the damage again increased.

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[9] Adviser, Coal and Iron Research Institute, Dortmund, Germany.

[10] H. Müller-Stock, "Der Einflusz dauerend und unterbrochen wirkender, schwingender Überbeanspruchung auf die Entwicklung des Dauerbruchs," *Mitteilungen* der Kohle-und Eisenforschung G.m.b.H. (1937).

[11] Professor of Mechanics, University of Wisconsin, Madison, Wis.

[12] *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 87 (1933).

The damage curves show that when a specimen is subjected to overstress the properties of the specimen are changed. Changes in the static properties may occur after a few cycles, and changes in the fatigue properties occur after more cycles. Different numbers of cycles at the same stress produce different changes. In general it may be said that every time a material is stressed it is changed slightly, and many cycles of stress may produce very appreciable changes.

COMPRESSIVE, IMPACT, AND OTHER MECHANICAL PROPERTIES OF NICKEL AND SOME OF ITS HIGH-STRENGTH, CORROSION-RESISTING ALLOYS

By Charles F. Catlin[1] and W. A. Mudge[1]

Synopsis

Compressive and impact properties have been determined for nickel, Inconel, and three types of Monel, and have been supplemented by corresponding tension and hardness data. The tension and torsion impact tests restrict suddenly applied stress to a definite area and give more comparable values than standard Izod and Charpy tests. The alloys are useful for vital parts of equipment requiring high strength and resistance to corrosion. The solid-solution or work-hardening alloys have true elastic properties in compression ranging from 15,000 to 83,000 lb. per sq. in. Similar values for "K" Monel, an age-hardening alloy, are from 23,000 to 98,000 lb. per sq. in.

Engineering progress continues to demand better materials of construction, particularly those which have high strength and hardness and are resistant to corrosion. Recent years have witnessed important developments of new alloys, a good proportion of which contains high percentages of nickel. Some of these have been solid-solution alloys, like Monel,[2] Inconel,[2] and stainless steels; others have been age-hardening alloys which have added the desirable feature of economy in construction because of a higher ratio of strength and hardness to weight. The aluminum-copper-nickel alloys recently described by Mudge and Merica,[3] one of which, "K" Monel,[2] is rapidly finding increased uses, are an excellent example of the latter group.

Industrial applications of alloys have generally been based upon tensile properties, hardness, and resistance to corrosion. Less attention seems to have been given to compressive and impact properties.

This study presents new data for high-strength, corrosion-resisting alloys of both the solid-solution and the age-hardening types. Compressive and impact properties have been determined for five important commercial materials, the nickel contents of which range from 65 to 99.5 per cent, and have been supplemented by corresponding tension and hardness values. This information should be useful for the design and construction of more efficient equipment.

Materials Used and Their Preparation

Chemical analyses of the materials are given in Table I.

"B" Monel is the regular Monel of industry. It is produced by calcination of a selected copper-nickel sulfide, reduc-

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[1] Research Metallurgist, and Works Metallurgist, respectively, Huntington Works, The International Nickel Co., Inc., Huntington, W. Va.

[2] Registered at U. S. Patent Office.

[3] W. A. Mudge and Paul D. Merica, "Aluminum-Copper-Nickel Alloys of High Tensile Strength Subject to Heat-Treatment," *Technical Publication 619*, Am. Inst. Mining and Metallurgical Engrs. (1935).

tion of the corresponding oxide with charcoal in acid open-hearth furnaces, duplexing and refining in basic electric furnaces, and casting into 14 by 14 by 60-in., 3200-lb. ingots.

TABLE I.—CHEMICAL COMPOSITION.

Alloy	Melt	Carbon, per cent	Sulfur, per cent	Silicon, per cent	Manganese, per cent	Iron, per cent	Copper, per cent	Nickel (+ Cobalt), per cent	Aluminum, per cent	Chromium, per cent
"B" Monel	M - 4052 - B	0.19	0.007	0.04	0.90	0.83	31.31	66.68	....	.....
"R" Monel	M - 4284 - R	0.06	0.041	0.01	1.04	1.13	31.13	66.57	....	.....
"K" Monel	K - 880	0.19	0.005	0.33	0.45	0.78	30.05	64.95	3.22	.....
Inconel	NX - 1396	0.06	0.008	0.17	0.11	5.48	0.10	81.04	....	13.01
"A" Nickel	N - 5604 - A	0.09	0.005	0.03	0.13	0.14	0.07	99.51	....	.....

TABLE II.—TENSILE AND COMPRESSIVE PROPERTIES (AVERAGE OF TRIPLICATE TESTS TO ± 50 LB. PER SQ. IN.).

Alloy	Condition	Tension							Compression			
		Yield Strength, lb. per sq. in.				Tensile Strength, lb. per sq. in.	Elongation in 2 in., per cent	Reduction of Area, per cent	Yield Strength, lb. per sq. in.			
		0.00 per cent Offset	0.01 per cent Offset	0.1 per cent Offset	0.2 per cent Offset				0.00 per cent Offset	0.01 per cent Offset	0.1 per cent Offset	0.2 per cent Offset
"B" Monel	Hot Rolled	34 650	36 650	40 000	40 650	83 750	39.5	67.5	30 150	33 050	37 350	38 050
	Cold drawn, 24 per cent reduction, stress relieved	69 000	75 000	84 650	86 650	97 250	27.0	66.4	50 800	57 950	69 650	80 750
	Cold drawn, annealed 1450 F., 3 hr.	24 000	27 650	32 000	33 350	78 350	44.0	65.9	16 150	19 200	25 650	28 250
"R" Monel	Hot Rolled	30 650	33 000	34 650	35 650	75 600	39.5	68.9	22 400	26 350	32 500	33 500
	Cold drawn, 24 per cent reduction, stress relieved	52 650	62 000	72 350	74 350	83 150	28.0	66.6	47 750	51 400	64 950	65 950
	Cold drawn, annealed 1450 F., 3 hr.	23 150	24 850	26 650	28 000	73 350	44.5	70.1	17 750	22 550	24 650	25 650
"K" Monel	Hot Rolled	28 650	41 000	46 100	47 000	99 900	42.5	63.7	23 250	34 000	38 150	40 350
	Hot rolled, age hardened	87 000	96 150	109 350	111 150	151 350	30.0	44.5	90 950	96 350	118 000	120 600
	Cold drawn, 24 per cent reduction, stress relieved	66 000	72 350	83 000	85 000	106 000	26.5	50.5	50 000	55 350	73 450	75 650
	Cold drawn, age hardened	93 350	103 000	115 650	119 650	157 650	22.0	37.5	97 900	102 150	119 000	121 250
Inconel	Hot Rolled	31 350	38 150	45 650	47 000	91 150	42.0	66.0	28 250	30 650	38 000	41 950
	Cold drawn, 24 per cent reduction, stress relieved	89 350	98 650	111 000	113 000	122 250	19.0	61.6	83 050	90 050	102 550	104 550
	Cold drawn, annealed 1750 F., 3 hr.	23 350	28 650	32 000	33 000	93 150	42.0	67.8	20 150	22 550	27 250	28 450
"A" Nickel	Hot Rolled	18 350	20 000	23 150	24 000	71 000	44.5	62.9	18 250	18 750	21 400	23 150
	Cold drawn, 24 per cent reduction, stress relieved	52 350	56 350	60 650	62 000	86 650	33.0	72.0	41 200	44 850	55 000	58 350
	Cold drawn, annealed 1350 F., 3 hr.	18 650	21 000	24 350	26 650	73 250	46.0	79.4	15 050	17 000	22 450	26 200

"R" Monel is a special product containing a small amount of sulfur, added to give free-machining properties. It has been available since 1931. Its production is similar to that of Monel.

"K" Monel[4] is a special product, first described three years ago.[3] It is produced by the addition of 2 to 4 per cent of aluminum to Monel. This gives the alloy age-hardening properties and makes it non-magnetic at temperatures as low as −110 F. Its production is otherwise similar to that of Monel.

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[4] U. S. Patents Nos. 1,572,744 (1926); 1,755,554, 5, 6 and 7 (1930).

Inconel is a nickel-chromium-iron alloy of high purity which has been developed within the past 6 yr. It is melted in basic electric furnaces, using electrolytic nickel and ferrochromium, and cast into 14 by 14 by 60-in., 3200-lb. ingots.

"A" Nickel is commercially pure malleable nickel.[5] It is produced by melting electrolytic nickel in acid open-hearth furnaces, and casting into 18 by 18 by 66-in., 7300-lb. ingots.

Processing:

Except for required differences in heating temperatures, the processing of all materials was essentially the same. The ingots were first mechanically overhauled by milling and chipping, then heated to temperatures varying from 2150 to 2350 F., depending upon the chemical compositions of the alloys, and forged into 8 in. square, 1200-lb. blooms. These were chipped to remove surface defects, reheated, and hot rolled to 3 in. square, 100-lb. billets.

Hot-Rolled Rods:

The billets were again chipped, reheated and hot rolled to 1-in. diameter rods. Following hot rolling "A" nickel and the solid-solution alloys—"B" and "R" Monel, and Inconel—were air cooled; "K" Monel was water quenched from 1450 F. to render it suitable for age hardening.

Cold-Drawn Rods:

The hot-rolled rods of "A" nickel and the three solid-solution alloys were annealed,[6] pickled, surface ground, and cold drawn to $\frac{7}{8}$ in. in diameter. Some rods were low-temperature annealed by heating at 575 F. for 3 hr.; others were soft annealed as shown in Table II. The hot-rolled "K" Monel rods were similarly processed except that the initial annealing was omitted because quenching after hot rolling had given sufficient softness for cold drawing.

Age Hardening:

These treatments were as follows:

(*a*) Hot-rolled "K" Monel . . . . 10 hr. at 1080 to 1100 F., furnace cooled.

(*b*) Cold-drawn "K" Monel . . . . 10 hr. at 1040 to 1060 F., furnace cooled.

METHODS OF TESTING

Compression, impact, tension, and hardness tests were made on the several alloys in the hot-rolled, cold-drawn and stress-relief annealed,[7] cold-drawn and soft-annealed, and, in the case of "K" Monel, the age-hardened conditions. The hardness tests represent at least ten determinations for each condition of each alloy; the other tests were made in triplicate.

The compression tests were made with standard cylindrical specimens[8] using a 100,000-lb. Baldwin-Southwark machine and a compression micrometer of 0.0002-in. sensitivity. The rate of loading was 0.01 in. per min. In some cases, the specimen length was increased to 5.50 ± 0.01 in., in order to accommodate a more sensitive instrument for checking proportional limit and Young's moduli data.

Four types of impact tests were performed with an Olsen machine: standard Izod,[9] standard Charpy,[9] Charpy tension impact, and Charpy torsion impact; the dimensions of specimens for the two latter are given in Fig. 1.

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[5] "Nickel," *Circular No. 100*, Nat. Bureau Standards (1921).

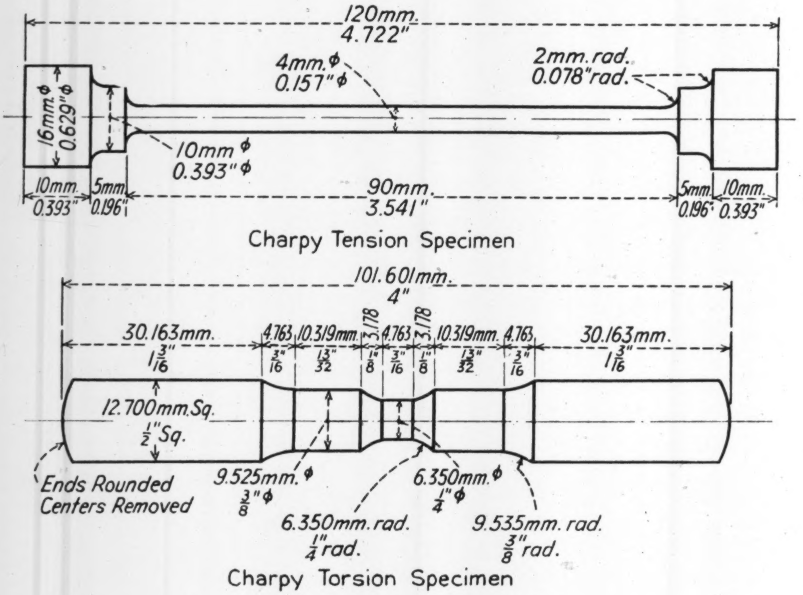
[6] C. A. Crawford, *Technical Publication 35*, Am. Inst. Mining and Metallurgical Engrs. (1927).

[7] This use of the term in the paper is not intended to mean a complete removal of all stress, but is a low-temperature treatment which develops maximum values in yield strength, 0.00 per cent set, as described by W. A Mudge in his discussion of the paper by Peter R. Kosting entitled "Stress-Relief Annealing High-Strength Monel Metal Plate," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 232 (1936).

[8] Tentative Methods of Compression Testing of Metallic Materials (E 9 – 33 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 959 (1933); also 1937 Book of A.S.T.M. Tentative Standards, p. 1499.

[9] Tentative Methods of Impact Testing of Metallic Materials (E 23 – 34 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part I, p. 1201 (1934); also 1937 Book of A.S.T.M. Tentative Standards, p. 1507.

The torsional impact test was performed with an Olsen Universal Impact Testing Machine which is provided with interchangeable pendulums, holders, and scales. True torsional stress is applied to a specimen which is held coaxially with the pivot axis of the hammer. The squared ends of the specimen (Fig. 1) fit snugly into grips, thereby eliminating slippage. One grip is integral with the center of rotation of the hammer; the other rotates in a ball-bearing set in one side of the rigid frame. The specimen therefore forms a straight coupling between the two grips. When the striking hammer reaches the bottom of its arc, the motion of the grip rotating in the frame is suddenly arrested by a suitable "pin and stop" arrangement; the other grip, being rigid with the hammer, continues to rotate. The inertia of the falling hammer produces the torsional force on the specimen. Torsion on the specimen does not begin until the hammer has achieved its maximum fall, so that the same impact force is brought to bear on the specimen each time. The angle of twist was measured to ±½ deg. with a suitable spirit-level protractor, using a line scribed along the specimen.



Charpy Tension Specimen

Charpy Torsion Specimen

FIG. 1.—Impact Specimens.

The tension tests were made with the same Baldwin-Southwark machine, using standard 0.505-in. specimens.[10] Stress-strain curves were obtained with an autographic recorder. The rate of loading was 0.01 in. per min. The values determined were the yield strengths at 0.00 per cent offset (proportional limit), at 0.01 per cent offset (proof stress), and at 0.1 and 0.2 per cent offsets; the tensile strength; the percentage elongation in 2 in. and the percentage reduction of area.

The hardness tests included Brinell[11]

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[10] Standard Methods of Tension Testing of Metallic Materials (E 8 - 36), 1936 Book of A.S.T.M. Standards, Part I, p. 833.

[11] Standard Methods of Brinell Hardness Testing of Metallic Materials (E 10 - 27), *Ibid.*, p. 817.

(3000-kg. load, 10-mm. ball), Rockwell[12] ("B" and "C" scales), and Vickers (30- and 50-kg. loads, diamond pyramid indenter, $\frac{2}{3}$-in. objective).

EXPERIMENTAL RESULTS

All data are summarized in Tables II and III and in Fig. 2.

DISCUSSION

Compression (Table II):

The values in compression for "K" Monel in the hardened conditions are slightly higher than the corresponding values in tension; the other compression values are lower than the tension values by the amounts shown in Table II. Loads greater than those necessary to produce 0.20 per cent offset gave progressive plastic deformation up to the limit of the testing machine, approximately 175,000 lb. per sq. in., and resulted in average area increases of 20 per cent and length decreases of 15 per cent with typical "barreling out" of the test pieces. None of the specimens fractured. The stress-strain curves showed reliably true elastic properties and no gradual deviations from the origin as is generally true for non-ferrous nickel alloys containing lower percentages of nickel. Within the limits of experimental error the moduli of elasticity in compression are the same as those in tension, that is, 26,000,000 for "B," "R," and "K" Monel, 30,000,000 for "A" nickel, and 31,000,000 for Inconel.[13]

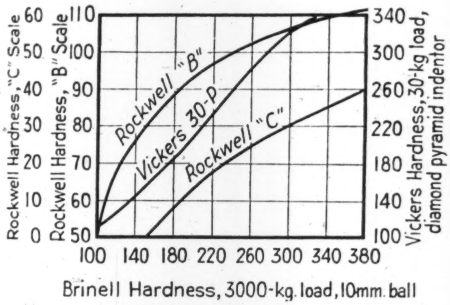

FIG. 2.—Hardness Conversion Curves.

"A" nickel has the lowest and Inconel the highest compressive values for the non-age-hardening materials; "R" and "B" Monel have intermediate values. The values of "K" Monel compare favorably with those of high-grade alloy steels.

The absence of any fracturing in the specimens is an important feature. Service overloads may, therefore, cause only permanent set and not sudden fracture. The former is less likely to result in serious damage to equipment.

Impact (Table III):

The standard Izod and Charpy tests are useful in showing that the metals studied have great toughness, but these tests do not provide in all cases an accurate measurement of toughness because the specimens of some of the metals have sufficient toughness to absorb the whole energy of the machine without breaking in two. For a more precise measurement of toughness, the tension impact test is more useful, as clean breaks are obtained in all cases.

Izod and Charpy specimens of "K" Monel, except in the hot-rolled condition, fractured completely, with clean breaks, similar to those of many ferrous alloys; all other specimens fractured partially[14] with sufficient bending to allow the hammer to pass and register values approximating, or equalling, the capacity of the machine. The majority of the partially fractured specimens showed plastic deformation over their entire length. Variations as great as 10 to 15 per cent were obtained by greasing the free end of an Izod specimen so as to decrease the friction caused by the passing of the striking hammer over the bent specimen. Some early irregularities and discrepancies in results may have been due to the presence of machining lubricant on the specimens; this may have introduced frictional energy differences which were erroneously attributed to the metals being tested. Attempts to make the Izod and Charpy tests more critical by varying the notches in the specimens were not satisfactory.

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[12] Standard Methods of Rockwell Hardness Testing of Metallic Materials (E 18 - 36), 1936 Book of A.S.T.M. Standards, Part I, p. 824 (1936).

[13] Carl Rolle, "Nickel and Nickel-Base Alloys in Wire and Rod Products," *Wire and Wire Products*, Vol. 12, pp. 640-653 (1937).

[14] N. B. Pilling, "Effect of Cold Working on the Izod Notched-Bar Impact Value of Monel Metal," *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part II, p. 576 (1932).

TABLE III.—IMPACT (AVERAGE OF TRIPLICATE TESTS TO ± 1 FT-LB.) AND HARDNESS (AVERAGE OF 10 TESTS) DATA.

Alloy	Condition	Brinell Hardness, 3000-kg. load, 10-mm. ball	Izod		Charpy		Charpy Torsion			Charpy Tension			
			ft-lb.[a]	ft-lb. per sq. in.	ft-lb.[a]	ft-lb. per sq. in.	ft-lb.[a]	ft-lb. per sq. in	Angle of twist, deg.	ft-lb.[a]	ft-lb. per sq. in.	Elongation in 3.54 in., per cent	Reduction of Area, per cent
"B" Monel....	Hot Rolled	145	>120	>954	232	1866	34	694	101½	138 (b)	7170	27.5	70.7
	Cold drawn, 24 per cent reduction, stress relieved	199	>120	>962	151	1201	39	788	98	96 (b)	4950	15.0	63.7
	Cold drawn, annealed 1450 F., 3 hr.	123	>120	>968	206	1649	30	599	102	129 (b)	6680	29.5	68.0
"R" Monel....	Hot Rolled	121	96	786	187	1512	30	606	100½	142 (b)	7460	27.0	68.4
	Cold drawn, 24 per cent reduction, stress relieved	180	99	792	140	1117	34	687	100½	90 (b)	4600	17.0	64.7
	Cold drawn, annealed 1450 F., 3 hr.	116	>120	>957	196	1584	30	606	102	148 (b)	7560	35.0	69.1
"K" Monel....	Hot Rolled	163	>120	>977	170	1366	36	727	100	180 (b)	9240	31.5	66.1
	Hot rolled, age hardened	299	40 (b)	323	61 (b)	484	42 (b)	833	40	167 (b)	8697	17.5	46.7
	Cold drawn, 24 per cent reduction, stress relieved	207	56 (b)	484	71 (b)	570	41	828	98	102 (b)	5230	17.5	55.3
	Cold drawn, age hardened	329	26 (b)	206	42 (b)	334	28 (b)	566	38	143 (b)	7367	14.5	40.2
Inconel........	Hot Rolled	159	>120	>952	191	1528	36	734	100	133 (b)	6830	30.0	67.2
	Cold drawn, 24 per cent reduction, stress relieved	210	>120	>980	151	1202	44	881	95	75 (b)	3880	9.5	57.4
	Cold drawn, annealed 1750 F., 3 hr.	136	>120	>950	230	1826	29	580	103½	169 (b)	8780	37.5	70.2
"A" Nickel...	Hot Rolled	107	>120	>932	200	1624	29	579	103½	98 (b)	4950	20.0	83.1
	Cold drawn, 24 per cent reduction, stress relieved	177	>120	>966	204	1645	35	702	102	88 (b)	4550	19.5	71.2
	Cold drawn, annealed 1350 F., 3 hr.	109	>120	>980	228	1823	29	586	103	113 (b)	5860	33.0	75.1

[a] (b) indicates specimen fractured completely.

The tension impact tests gave clean breaks in every case, and permitted accurate calculations of ultimate fracture strength in foot-pounds per square inch, percentage elongation and percentage of reduction of area, as well as foot-pounds of impact.

The torsion impact tests produced fractures in "K" Monel only. However, the method of holding the specimen permitted no variations in results due to friction of the striking hammer. From 97 to 99 per cent of the twist was confined to the section of minimum area. Attempts to produce fractures in "A" nickel and in the solid-solution alloys by reducing this area 75 per cent were not successful.

The tension and torsion impact tests have an advantage over the Izod and Charpy tests when used on metals of high toughness, inasmuch as they permit suddenly applied stress to be restricted to a definite area, rather than to extend over a large region of the specimen. More comparable values are obtained from the tension and torsion impact tests.

Further work is in progress on this phase of the subject to determine the best method of numerically expressing impact data and coordinating these with other mechanical properties.

Tension (Table II):

These are typical of standard values for the alloys[15] in the conditions tested, especially those recently published by Rolle.[13]

Hardness (Table III):

These values agree with previous results and require no special comment. Figure 2 has been included for approximate conversion purposes; these curves are equally applicable to all of the alloys and conditions investigated.

Comparison of Alloys:

Too strict a comparison cannot be made because each alloy has found, and is continuing to find, definite applications in which it gives most efficient service. All of these alloys are malleable and may be fabricated by hot and cold working and machining.[16]

"B" Monel[17] and "A" nickel are well established. Detailed summaries of their strength and corrosion-resisting properties have been given by McKay and Worthington.[18]

"R" Monel is a free-machining alloy and is particularly adapted to the manufacture of automatic screw machine products. It has the same corrosion resistance as Monel, which is somewhat stronger and is therefore recommended in preference to "R" Monel where strength is the important factor. However, the tensile strength and elongation of "R" Monel considerably exceed the minimum requirements for structural carbon steel for bridges and buildings as defined by A.S.T.M. Specifications A 7 - 36 and A 9 - 36.[19]

Inconel[20] was developed primarily for corrosion resistance to tarnishing and service in the food industries and has given good results in these applications. Because of its freedom from scaling and intercrystalline attack it is suitable for use at elevated temperatures.

Inconel and "K" Monel[21] are nonmagnetic to −40 F. and −110 F., respectively. This property enhances their usefulness in certain services, such as aeronautical instruments where the absence of magnetic effects is essential.

The remarkably high strength and hardness which may be developed in "K" Monel, by suitable age-hardening treatments, place this alloy in the front rank of new materials of construction. It has been found to give excellent service in pump rods, propeller shafts, and steam-valve trim uses.

CONCLUSIONS

1. Compressive and impact properties, supplemented by corresponding tensile and hardness data, have been determined for nickel, Inconel, and three grades, or types, of Monel.

2. The tension and torsion impact tests have an advantage over the Izod and Charpy tests, when used on metals of high toughness, because they permit suddenly applied stress to be restricted

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[15] W. A. Mudge and L. W. Luff, "Some Mechanical Properties of Nickel, Manganese-Nickel and Copper-Nickel Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 28, Part II, p. 278 (1928).

Peter R. Kosting, "Stress-Relief Annealing High-Strength Monel Metal Plate," *Proceedings*, Am Soc. Testing Mats., Vol. 36, Part II, p. 222 (1936).

[16] "Machining Monel, Nickel and Inconel," *Technical Bulletin T-12*, The International Nickel Co., Inc. (1937).

[17] "Engineering Properties of Monel," *Technical Bulletin T-5*, The International Nickel Co., Inc. (1936).

[18] Robert J. McKay and Robert Worthington, "Corrosion Resistance of Metals and Alloys," ***Monograph No. 71***, Am. Chemical Soc. Series (1936).

[19] Standard Specifications for Steel for Bridges (A 7 - 36), 1936 Book of A.S.T.M. Standards, Part I, p. 1.

Standard Specifications for Steel for Buildings (A 9 - 36), *Ibid.*, p. 9.

[20] "Properties and Uses of Inconel," ***Technical Bulletin T-7***, The International Nickel Co., Inc. (1937).

[21] "Engineering Properties of 'K' Monel," ***Technical Bulletin T-9***, The International Nickel Co., Inc. (1937).

to a definite area rather than to extend over a large region of the specimen, thereby giving more comparable values.

3. The usefulness of these alloys for vital parts of equipment is indicated by the properties determined. "A" nickel and the solid-solution, or work-hardening, alloys have true elastic properties in compression ranging from 15,000 to 83,000 lb. per sq. in. Similar properties of the age-hardening alloy are from 23,000 to 98,000 lb. per sq. in.

DISCUSSION

MR. PETER R. KOSTING[1] (*presented in written form*).—When studying metals for purposes of comparison and of engineering design, the necessity of making tests at various velocities of impact was first pointed out by Sir Robert Hadfield in 1906, and of recent years H. C. Mann has established an understandable basis for such work. He used tension impact specimens. Watertown Arsenal has not made variable velocity impact tests of high nickel alloys. However, with reference to K monel and even B monel, limited tests of the nature described by Mann[2] have shown that the energy value obtained from the load-elongation curve (static test) is more than the energy value obtained in the impact (dynamic) test when the velocity of impact was about 28 ft. per sec. This indicates a falling off of impact energy at some velocity of impact less than 28 ft. per sec. Direct observations of the transition velocity should therefore be made.

On page 274 of the paper, it is mentioned that the tension impact test permitted calculation of ultimate fracture strength in foot-pounds per *square inch*. In 1937, Mann[3] showed that the impact energy for the steels tested is dependent upon the *volume* of material participating in the absorption of energy. The tension impact specimen within reasonable limits of size and with square shoulders permits the accurate measurement of that volume. It follows that with reference to the general type of specimen shown on the top of Fig. 1, if the length were halved and the cross-sectional area doubled, the same impact value would be obtained because the volume is the same. For the *same* length, the impact value will change proportionally as the cross-sectional area is changed because under this limitation the volume changes proportionally as the cross-sectional area.

In ordnance, the transverse properties of metals are important observations, and consequently longitudinal and transverse tests are made especially when checking a new material. The following shows a comparison of R monel in the transverse and longitudinal directions, test specimens being taken at 50 per cent radius of a 4 by 4-in. billet.

Sulfur, per cent	Direction	Yield Strength, lb. per sq. in.			Tensile Strength, lb. per sq. in.	Elongation, per cent	Reduction of Area, per cent	Impact[a] Tension Notch, ft-lb.
		0.00 per cent Offset	0.05 per cent Offset	0.20 per cent Offset				
0.027	Longitudinal	29 500	33 400	36 100	81 700	45.5	69.5	67
	Transverse	28 500	33 400	35 700	80 500	40.0	51.0	48
0.037	Longitudinal	28 500	34 500	36 000	72 300	45.0	71.5	59
	Transverse	29 000	34 300	36 100	70 500	33.0	38.0	38

[a] Specimen described in Federal Specification QQ-M-151a.

It is to be observed that the transverse properties of this "free machining" quality of monel have held up remarkably well for these size billets with these contents of sulfur. Of particular note are the values for elongation and reduction of area and impact, and how the effect on the transverse values of these properties increases as the sulfur content increases.

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[1] Chemical Engineer, Watertown Arsenal, Watertown, Mass.

[2] H. C. Mann, "The Relation Between the Tension Static and Dynamic Tests," *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part II, p. 323 (1935).

[3] H. C. Mann, "A Fundamental Study of the Design of Impact Test Specimens," *Proceedings*, Am. Soc. Testing Mats., Vol. 37, Part II, p. 102 (1937).

MR. H. C. MANN[4] (*presented in written form*).—I should like to point out that details of shape of notch should be given. Shape at base of notch governs volume which can be greatly varied by slight dimensional changes.

Results from specimens which are not completely fractured are of little value. Values in Table III of the paper are high for a sharp notch specimen.

One would expect erratic results from double necking in the tension impact specimen used (Fig. 1). We have been unable to avoid this unless the gage section is tapered toward the middle.

MR. W. A. MUDGE.[5]—I appreciate very much the comments which have come from the Watertown Arsenal, and, as I stated in the formal print of the paper and repeated in my summary, the impact data which we have determined, with respect to Charpy tension and Charpy torsion, are new and we are offering what we have obtained thus far for such use as the data may serve.

None of our tension impact specimens was tapered. We observed no cases of double necking, although approximately one-third of the specimens fractured about $\frac{1}{2}$ to $\frac{3}{4}$ in. from the fillet.

We fully appreciate the fact that we have not necessarily arrived at the correct specimens to use for these high nickel, and therefore very tough, alloys. I personally hope that when we shall have completed the investigation which is now under way, we shall be able to report as fine a series of results as has come from Watertown Arsenal.

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[4] Senior Materials Engineer, Watertown Arsenal, Watertown, Mass.

[5] Works Metallurgist, Huntington Works, The International Nickel Co., Inc., Huntington, W. Va.

AN ARC SOURCE FOR QUANTITATIVE SPECTRAL ANALYSIS

By C. J. Neuhaus[1]

In spectral analysis the sample specimen to be analyzed is vaporized and its atoms are excited in either a flame, an arc, or a spark, resulting in the radiation of light that contains the wavelength patterns peculiar to the emitting elements.

The choice of the type of source—flame, arc, or spark—depends on a number of conditions. In quantitative spectral analysis the source selected must certainly fulfill one condition—it must permit reproducible results. Generally the spark method is recognized as the more reliable one in this respect; however, conditions may arise to jeopardize the applicability of this method of excitation. The low intensity of the lines of some elements in the spark spectrum when present at low concentrations is illustrative. With the flame method ruled out because of its limited scope of determinable elements, the arc method remains. Other restrictive conditions that led to the arc method, that will be described, are the following. Solution and solid electrode arcs were out of the question, since they either required too much time or too much material to prepare. The size of the specimen must be small so as to facilitate its preparation from almost anything, such as sheet, wire, tubing, and strip. The globule method had not proved itself very reliable as to reproducibility; therefore it was ruled out.

Based on these points the arc arrangement described below and shown on Fig. 1 was designed.

The specimen to be analyzed is made the cathode; this is normally the lower electrode. The anode or the upper electrode consists of a sufficiently pure metal, preferably the main component present in the specimen. The cross-section of the specimen is 0.03 by 0.13 in. The minimum length that can be accomodated in the specimen holder is 0.25 in. The upper electrode has a cross-section of 0.25 by 0.25 in. Its lower end is cut to a slender cone with a point of approximately 0.03 in. in diameter. The specimen is clamped in a copper holder with only 0.18 in. of the former's length protruding from the holder. The copper clamp in turn is fastened by means of a set screw to a copper radiator, arranged so that the long side of the specimen's cross-section is in the optical axis of the spectrograph. The function of the copper radiator is to dissipate the heat generated by the arc. The arc gap is made 0.13 in. and the arc is operated at about 1.8 amp. on a 220-v. direct current line. A series resistance of 107.5 ohms and an inductance were connected in series with the arc.

To indicate the accuracy and precision of this method, several spectra of three monel samples, containing, respectively, 0.016, 0.049, and 0.080 per cent aluminum, were taken on three different plates using a large Bausch & Lomb Littrow type spectrograph. The arc was kept burning for 120 sec. before the actual exposure of 30 sec. was made. The spectra were taken on Eastman spectrographic plates, type IV-O special, which were developed for 2 min. at 65 F. in the hydroquinone caustic developer, Eastman formula D9. The blackenings of an aluminum line, Al 3961.537, and a nickel reference line, Ni 3793.60, were then measured by means of a Bausch & Lomb density comparator and the logarithms of the ratios of the comparator deflection values plotted against the logarithms of the aluminum concentrations.

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[1] Spectrographic Laboratory, The International Nickel Co., Inc., Huntington, W. Va.

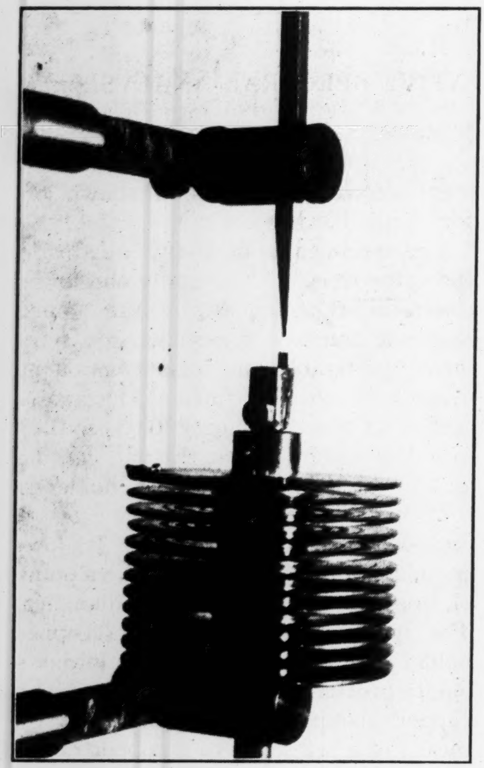

FIG. 1.—Arrangement of the Arc.

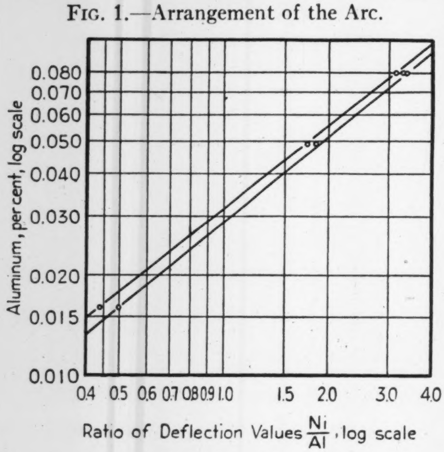

FIG. 2.—Deflection Values Obtained from Seven Different Spectra of Three Analyzed Samples, Plate No. 1.

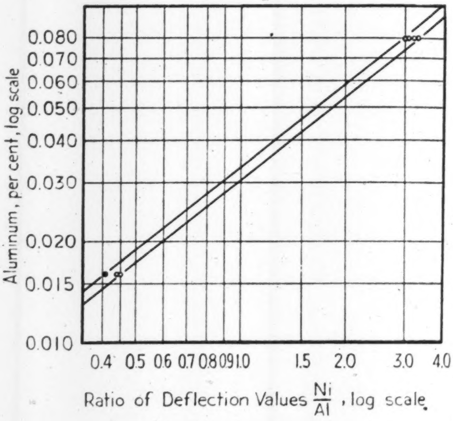

FIG. 3.—Deflection Values Obtained from Seven Different Spectra of Two Analyzed Samples, Plate No. 2.

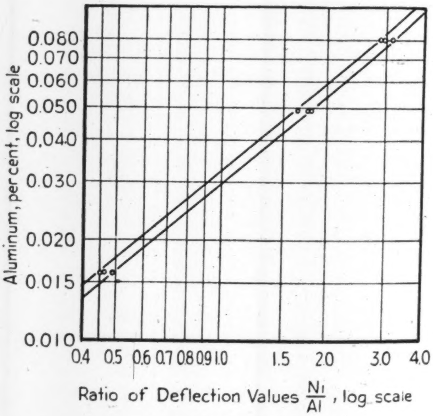

FIG. 4.—Deflection Values Obtained from Nine Different Spectra of Three Analyzed Samples, Plate No. 3.

The results are compiled in Table I and are also shown as graphs on Figs. 2, 3, and 4.

The spread of the points on the graph has been delimited by lines. The vertical displacement of these lines should permit a conclusion as to the accuracy in terms of percentages. Measurement indicates the total error to be of the order of 10 per cent of the content under consideration.

Part of the error can be traced to the density comparator. It is hoped to eliminate or minimize this error by installing a different photoelectric cell, soldering all connections in the cell circuit, and motorizing the plate transport across the slit of the density comparator.

With all solid electrode methods, including the one described, only a relatively small amount of the sample material is volatilized during an exposure. Consequently there is danger of not obtaining a representative sample. This danger, of course, is especially acute if segregation exists. If there is suspicion that this condition prevails, several specimens of the sample may be taken and the results compared.

In the particular analysis mentioned, the arc burned very steadily, moving slowly forward and backward over the specimen and consuming about 4 to 5 mg. per min. Current fluctuations were observed to be limited to about ±0.05 amp.

TABLE I.—DETERMINATION OF RATIO OF DEFLECTION VALUES FROM THE DIFFERENT SPECTRA BY MEANS OF A BAUSCH & LOMB DENSITY COMPARATOR.

Plate	Spectrum	Specimen	Al Concentration, per cent	Deflection Values		Ratio of Deflection Values Ni/Al	
				Ni 3793.60	Al 3961.537		
No. 1..	No. 1...	No. 10...	0.016	7.3	16.6	0.44	See Fig. 2
	No. 2...	No. 11...	0.049	9.4	5.4	1.74	
	No. 3...	No. 12...	0.080	6.3	2.0	3.15	
	No. 4...	No. 10...	0.016	7.9	15.7	0.50	
	No. 5...	No. 11...	0.049	6.4	3.5	1.83	
	No. 6...	No. 12...	0.080	6.3	1.9	3.32	
	No. 7...	No. 12...	0.080	6.1	1.8	3.39	
No. 2	No. 1...	No. 10...	0.016	9.4	20.7	0.45	See Fig. 3
	No. 2...	No. 10...	0.016	7.9	18.1	0.44	
	No. 3...	No. 10...	0.016	7.3	17.8	0.41	
	No. 4...	No. 12...	0.080	7.2	2.4	3.00	
	No. 5...	No. 12...	0.080	9.3	2.9	3.20	
	No. 6...	No. 12...	0.080	7.3	2.4	3.04	
	No. 7...	No. 12...	0.080	8.9	2.7	3.30	
No. 3..	No. 1...	No. 10...	0.016	7.0	15.4	0.45	See Fig. 4
	No. 2...	No. 10...	0.016	6.6	14.8	0.45	
	No. 3...	No. 10...	0.016	9.2	18.8	0.49	
	No. 4...	No. 11...	0.049	8.4	5.1	1.65	
	No. 5...	No. 11...	0.049	8.3	4.6	1.80	
	No. 6...	No. 11...	0.049	8.8	5.0	1.76	
	No. 7...	No. 12...	0.080	8.1	2.6	3.12	
	No. 8...	No. 12...	0.080	8.6	2.9	2.96	
	No. 9...	No. 12...	0.080	7.5	2.6	2.88	

DISCUSSION

Mr. H. V. Churchill[1] (*presented in written form*)—Before discussing this interesting paper, the writer wishes to ask these questions:

1. Were separate electrodes used for the preparation of each plate?

2. In applying this method to unknown samples, how many spectra does the author prepare? If only one spectrum is used, how does the spectrographer know whether the ratio obtained is a minimum or a maximum for the specific aluminum concentration present?

This paper is not subject to the criticism that is applicable to many contemporary papers on spectrographic analysis, in that the method is given for a specifically limited range of concentration of element to be determined. All too many of those contemplating the use of quantitative spectrography are mislead as to the generality of applicability of specific methods which, as a matter of fact, are applicable only to a limited range of concentration.

This paper is of interest because it shows some of the advantages of spectrographic analysis. It is far simpler than equally precise chemical procedures, and is far less time-consuming.

Fundamentally the quantitative analyses of materials must depend upon chemical procedures. As a rule, chemical methods are applicable over wider ranges of composition than are spectrographic methods. However, the method under discussion clearly reveals how the spectrograph can be economically applied to routine testing in a limited range of composition. Usually, if the range of expected composition is known, satisfactory spectrographic methods can be worked out for many elements.

Sources of spectral excitation constitute one of the most important problems confronting the student of quantitative spectrography. This paper shows that despite the known disadvantages of the arc in quantitative procedures, there exist certain fields where the advantages outweigh the difficulties. The fact remains, however, that spark sources are far more valuable than arc sources for most spectrographic work of a quantitative nature. While the modern tendency is towards the use of controlled high voltage sparks, it is refreshing to have our attention called to the utilization of a simple arc.

It is interesting to note that the author ascribes much of the error in his results to the behavior of the densitometer used. This conforms to the experience of many workers in this field, that there is considerable improvement to be made in densitometers for routine use by those who need such instruments for quantitative spectrography. This is a fertile field, and it is to be hoped that helpful developments in this field will soon be made.

Mr. T. A. Wright.[2]—In the Society's Committee E-2 on Spectrographic Analysis, our minds run somewhat in the same channels, and Mr. Churchill has well covered, and better covered some of the points I have in mind, but I do want to express my gratification that we have a paper here on the very important matter of excitation which is so often passed over lightly or assumed to cover too many generalities. I was interested as to whether this scheme could be applied to other determinations or other elements than nickel and I should like to ask that question of Mr. Neuhaus and as to what goes on. I was also interested in the design of the copper radiator, and, in particular, the low amperage used. In connection with my first question as to the other elements that might be determined by a similar procedure, I was a little concerned as to some of those in view of the fact that apparently the first few minutes of exposure are ignored. Of course as to certain of these elements, one could not do that, and I would like to ask how it was determined that a 120-sec. prior exposure should be made before taking the spectrum?

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[1] Chief Chemist, Aluminum Research Laboratory, Aluminum Company of America, New Kensington, Pa.

[2] Technical Director and Secretary, Lucius Pitkin, Inc., New York City.

MR. W. B. COLEMAN.[3]—I should like to ask Mr. Neuhaus just what difficulties he encountered in the use of the spark. It seems that there is a great deal of controversy between the arc and the spark as to which source should be used in the determination of metals, and since he stated that the spark did not seem to work out too well he probably got into difficulties somewhere. I would like to know.

MR. C. J. NEUHAUS[4] (*author's closure*).—Replying to Mr. Churchill's first question, I wish to say that the three plates I have shown were made for experimental purposes. The electrodes were about $\frac{13}{16}$ in. long. For each exposure, a length of $\frac{3}{16}$ in. was consumed; hence, after four exposures the preparation of a new electrode became necessary. For instance, of the specimen No. 10, a new electrode was employed when taking the spectrum No. 3 on plate No. 2.

Replying to the second question, we generally duplicate all exposures of the standards as well as of the unknown samples.

Concerning Mr. Wright's question; the preliminary arcing time of 120 sec. was determined by taking 10 successive exposures of 30 sec. each with 5-sec. intervals while the arc was kept burning. Calculation of the nickel over aluminum ratios showed that they became approximately constant after about 120 sec. For other elements than aluminum, this preliminary arcing time may not be desirable. I have not made any experiments using this method with other base metals than nickel alloys.

Replying to Mr. Coleman's question; in quantitative analysis we prefer to use the spark; however, sometimes a suitable pair of analysis and reference lines is not available. This is especially liable to occur when determining elements present at low concentrations. In such a case the arc may offer a solution.

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[3] President, W. B. Coleman and Co., Philadelphia, Pa.

[4] Spectrographic Laboratory, The International Nickel Co., Inc., Huntington, W. Va.

SECONDARY RADIATION IN THE RADIOGRAPHY OF ALUMINUM, STEEL, AND LEAD

By Herman E. Seemann[1]

The detrimental influence of secondary X-radiation[2] on the quality of a radiograph is a well-known effect. Some appreciation of its magnitude may be obtained by comparing an ordinary radiograph with one taken with the aid of a Potter-Bucky diaphragm, lead-foil screen, or, under certain conditions, a narrowly defined X-ray beam. There is usually a very noticeable increase in the amount of detail made visible, although none of these methods completely removes the diffuse secondary radiation. This radiation obscures detail in the same manner that detail would be lost in a pictorial photograph if the film were given a uniform exposure to light before use. There would be an *apparent* increase in the sensitivity or speed of the film because it would naturally take less additional exposure to produce a given density than without the preliminary flash. However, the picture *quality* would be disappointing and its character described as lacking in contrast. The same type of result is obtained in a radiograph because the secondary radiation produces a more or less uniform exposure superposed on the primary image.

An analogy to the X-ray problem is the appearance of a distant object viewed through a light fog or haze. The eye may receive plenty of light but much of it has been scattered by the fog which reduces the contrast in the useful image on the retina.

The problem may be considered from the standpoint of the characteristic curve of the radiographic film. Curve 0, Fig. 1, is a typical relation between density and X-ray intensity. It is assumed in the scale of abscissae that there are various thicknesses of metal which transmit these arbitrary units of intensity. The slope of this curve at any point is a quantitative indication of the contrast or ability to show faint differences in incident X-ray intensity. Under fixed conditions of definition, adequate illumination, etc., the human eye can distinguish a certain fixed difference in density. Assuming that a given defect in a piece of steel represents a certain percentage increase in transmitted intensity, it is apparent that in moving up this curve to regions of higher contrast, this defect will come into regions of better visibility (up to a certain limit), or, smaller defects representing less change in transmitted intensity may be visible on the steep part of the curve and not at all visible in regions of less slope. This curve 0 is the kind of film characteristic which would result if there were no secondary radiation. It is, of course, a property of the film itself and its conditions of development. Suppose that in obtaining this curve a uniform intensity amounting to 16 arbitrary units is superposed. This gives curve 1, and if it is assumed that the primary radiation in the radiograph is also about 16 units, primary and secondary radiation occur in equal amounts. For the same density on the film the contrast has been reduced with this addition of secondary radiation. Curve 2 represents 32 units of intensity added to the original, or two parts of secondary radiation to one part of primary. Similarly, curve 3 represents 48 units of secondary added to the original curve 0. These are calculated results but they are based on definite knowledge of the photographic characteristic curve. As will be seen later, it is not an extreme case to assume, as in curve 3, three times as much secondary radiation as primary. These curves show quantitatively how the photographic film *appears* to have a lower contrast than would be found for it in laboratory tests. Curves 1, 2, and 3, therefore, are not unique film characteristics but are the *apparent* characteristics which exist under various conditions of radiography.

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[1] Physicist, Kodak Research Laboratories, Rochester, N. Y.

[2] Secondary X-radiation includes fluorescent X-rays, which are characteristic of the irradiated substance, scattered rays of the same wave length as the primary, and those which have experienced the Compton shift. The fluorescent X-rays from aluminum and iron are of such long wavelengths that their penetration of an ordinary cassette front is negligible. The K fluorescence from lead is, however, fairly penetrating.

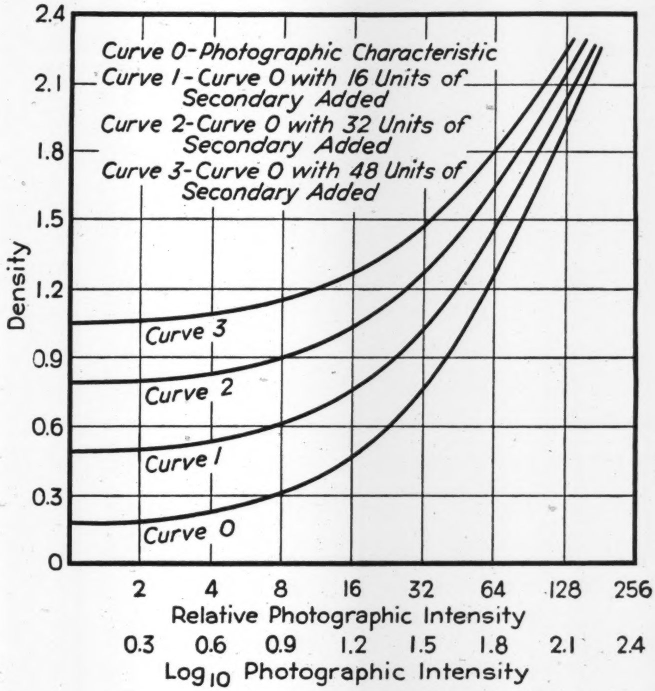

FIG. 1.—Effect on Photographic Characteristic of Addition of Secondary Radiation.

The problem of decreasing apparent contrast with increasing secondary radiation may be approached from a slightly different standpoint. Consider, for example, two adjacent areas in a radiograph, one area representing a flaw, and the other, sound material. Suppose the X-ray intensity under the flaw is 5 units and in the surroundings, 1 unit. Arbitrarily assign to this a contrast of 5 divided by 1, or 5. Now, add one unit of diffuse secondary radiation to both. The contrast changes to 6 divided by 2, or 3. Adding another unit of secondary, the ratio becomes 7 divided by 3 or $2\frac{1}{3}$. Another unit added yields the ratio 8 divided by 4, or 2 (see Table I). In other words, the same quantity is being added to the numerator and denominator of a number greater than unity and, therefore, the number representing contrast is diminishing.

There is considerable information available in the literature of physics on the subject of the different kinds of secondary effects produced by X-rays. The experiments performed in this field, however, have been approached from the standpoint of understanding the mechanism of production of secondary radiation or its use as a tool in the study of atomic structure. From the standpoint of industrial radiography, most of these experiments must be considered as too idealized for *direct* application to this problem. For example, monochromatic radiation is frequently used but heterogeneous radiation is all that is available for radiography. Consequently, any effect which varies with wave length cannot be satisfactorily interpreted when using heterogeneous radiation unless the spectral distribution is known. This is ordinarily not the case in practical radiography. The most satisfactory approach to the problem seems to be the arrangement of experimental conditions to approximate those of radiographic practice while still maintaining precise control over the various factors so that the results can be applied to radiographic technique if desired.

TABLE I.—ASSUMED INTENSITIES IN A RADIOGRAPH ILLUSTRATING THE DECREASE OF CONTRAST WITH INCREASE IN DIFFUSE SECONDARY RADIATION.

PRIMARY INTENSITY THROUGH FLAW	PRIMARY INTENSITY THROUGH SURROUNDINGS	SECONDARY INTENSITY	CONTRAST
5	1	0	$\frac{5+0}{1+0} = 5$
5	1	1	$\frac{5+1}{1+1} = 3$
5	1	2	$\frac{5+2}{1+2} = 2\frac{1}{3}$
5	1	3	$\frac{5+3}{1+3} = 2$
5	1	4	$\frac{5+4}{1+4} = 1\frac{4}{5}$

The experiments reported here were undertaken with the hope that they might increase our knowledge of seconary radiation in radiography. Apparently, there has been no quantitative work done on industrial materials from this standpoint. Wilsey[3] made a thorough study of the scattered radiation from water in 1921. This material was chosen because its radiographic characteristics are similar to those of human tissue and therefore the results would be applicable in medical radiography.

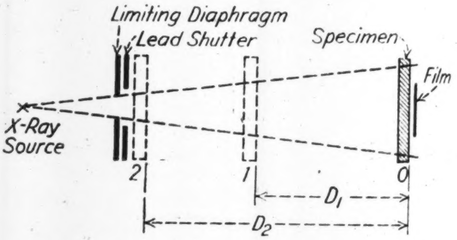

FIG. 2.—Schematic Diagram of Method for Measuring Secondary Radiation.

Auxiliary diaphragms for elimination of extraneous secondary radiation not shown.

Accurate estimation of the proportion of secondary radiation in an actual radiograph is difficult, if not impossible. A given density always represents the sum of primary and secondary radiation and there is no convenient way of separating the two quantities. A laboratory procedure[4] which is somewhat tedious but makes such a separation possible is illustrated diagrammatically in Fig. 2. The X-ray beam is confined to a fairly narrow cone by the limiting diaphragm. An exposure is then made with the specimen at the position represented by a distance D_1 from the film. Another exposure is made through the specimen at a distance D_2 from the film. In both of these cases, the primary beam receives the same filtration but the intensity of the secondary radiation at the film is different. Since there is the same radiation flux through the specimen in all positions, the *total* secondary radiation emitted will be the same in all positions. Therefore, the intensity of the secondary radiation at the film will be inversely proportional to the square of the distance D. Of course, it is assumed that the area of the specimen through which the X-rays pass is small compared to the distance. In other words, for practical purposes it may be considered as a point source. This assumption has been justified experimentally[4] for the conditions prevailing. From the data thus obtained, the intensity of the primary radiation may be calculated by extrapolating the results to a distance D equal to infinity. If the specimen is now placed close to the film as it would be in making a radiograph, the film will record the primary radiation plus the full value of the secondary radiation. If the intensity of the primary beam is subtracted from this total intensity, the secondary intensity is obtained. Thus, it is possible to calculate the amount of secondary radiation per unit of primary radiation transmitted by the specimen.

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[3] R. B. Wilsey, "The Intensity of Scattered X-rays in Radiography," *American Journal of Roentgenology*, Vol. 8, No. 6, June, 1921, p. 328.

In the following experiments, the distance from the X-ray focal spot to the film was 141 cm. The limiting diaphragm was located 33 cm. from the focal spot and consisted of a 4-cm. square hole in a lead plate. This gave an area a little over 17 cm. square at the film. While not large enough to yield the full amount of secondary radiation that would be obtained in a radiograph of large area, tests indicated that the error could be of the order of 10 per cent for thick sections but negligible for thin sections. The experiments of Wilsey[3] and of Müller[5] also indicate that no great increase in secondary radiation would be observed for irradiated areas larger than those used in this work and for these thicknesses. For the purposes of the present experiments, an even larger error in this factor would be permissible.

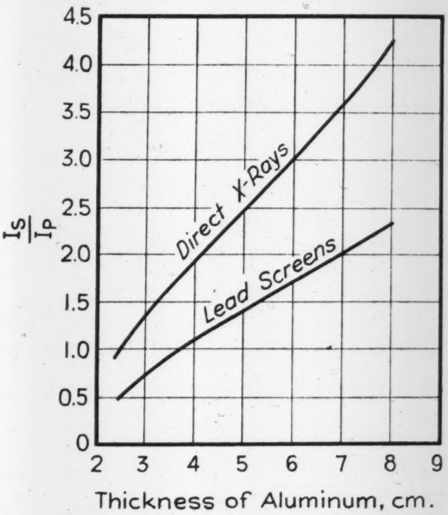

FIG. 3.—Proportion of Secondary Radiation as a Function of Thickness of Aluminum at 140 kv. Constant Potential.

For the sake of experimental convenience, Eastman Ultra Speed film was used in preference to No-Screen film in these experiments. The lower contrast of the Ultra Speed film made it possible to render satisfactorily the densities obtained both with and without lead screens at the same exposures. For practical X-ray radiography, however, the higher speed and contrast of the No-Screen film is to be preferred for work where the exposures do not require the use of calcium tungstate screens. Three materials were used: aluminum, cold-rolled steel, and lead. Aluminum was chosen to represent commercial aluminum and its alloys; cold-rolled steel represents iron and its alloys; and lead represents the heavy elements which though not commonly used in large proportions in commercial alloys, may be of interest in helping to understand the general problem.

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[4] Herman E. Seemann, "Secondary Radiation Intensity as a Function of Certain Geometrical Variables," *American Journal of Roentgenology*, Vol. 39, No. 4, April, 1938, p. 628.

[5] E. A. W. Müller, "Exposure Tables for Industrial Radiography and Their Application," *Fortschritte auf dem Gebiete der Röntgenstrahlen*, Vol. 56, No. 5, November, 1937, p. 676.

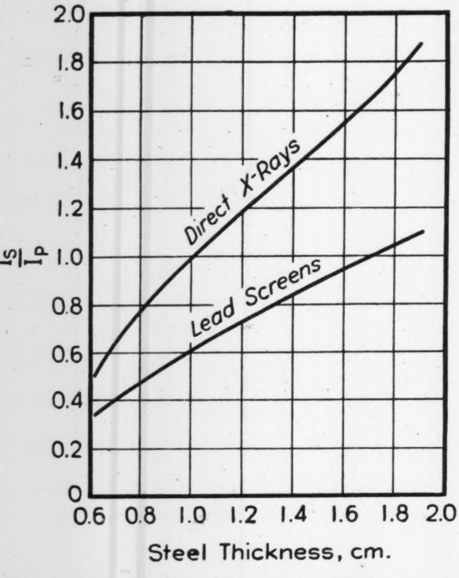

FIG. 4.—Proportion of Secondary Radiation as a Function of Thickness of Cold-Rolled Steel at 200 kv. Constant Potential.

Three different thicknesses of each material were used and a curve plotted of the logarithm of total intensity *versus* thickness, and the logarithm of primary intensity *versus* thickness. Such total intensity curves are almost straight lines, while the primary intensity curves have a greater curvature because of the absence in the latter of secondary radiation. This, plus the fact that the greater curvature is naturally in the high-intensity region, justifies interpretation of curves with only three data points. From these the following curves were calculated.

Figure 3 shows the proportion of secondary radiation as a function of thickness of aluminum at 140 kv. constant potential. Let I_s be the intensity of the secondary radiation and I_p, the intensity of the primary radiation. Therefore, I_s divided by I_p is the amount of secondary radiation per unit of primary intensity forming the useful image. In all of these experiments, simultaneous exposures were made with and without lead screens in order to study their characteristics. It is apparent from the ratios of ordinates of these two curves that the 0.014-cm. lead screens used removed almost one-half of the secondary radiation.

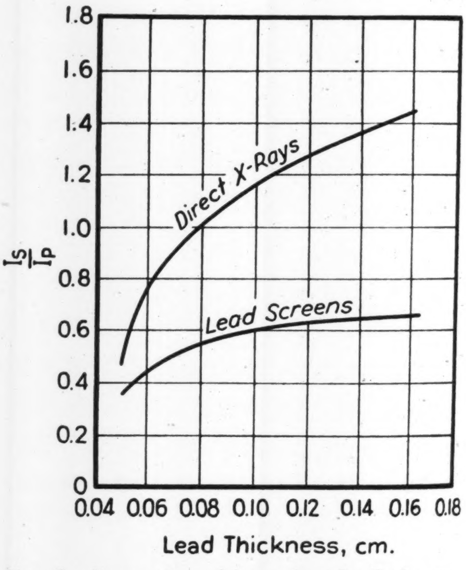

FIG. 5.—Proportion of Secondary Radiation as a Function of Thickness of Lead at 200 kv. Constant Potential.

Figure 4 shows the proportion of secondary radiation in the radiography of cold-rolled steel at 200 kv. constant potential. Again, it is found that the same lead screens remove almost one-half of the secondary radiation.

Similar curves for the radiography of lead are shown in Fig. 5. Lead was included because it is a convenient representative of the heavy elements and not because radiographic inspection of this very opaque substance is common.

Some estimate of the accuracy of the determination of I_s/I_p may be obtained from the following considerations: the total intensity, I_t, and the primary intensity transmitted, I_p, are measured quantities. The secondary intensity, I_s, is found from the relation $I_t = I_p + I_s$. But the proportion of secondary radiation is given by $I_s/I_p = I_t/I_p - 1$. If, for example, I_s and I_p are equal, a 1 per cent error in the determination of both I_t and I_p may result in a 4 per cent error in I_s/I_p. When I_s/I_p is larger, the error will be less, and *vice versa.* For a given position and thickness of specimen (Fig. 2), three exposures were made and three densities read on each film, making nine readings in all. These were averaged for one point on the curves from which the I_s/I_p *versus* thickness curves were calculated. The average deviation from the mean of such a set seldom exceeded 3 per cent. This would result in about 12 per cent error in I_s/I_p. Repetitions of these experiments indicated that experimental errors of this magnitude must be expected. Although photographic measurements, in general, are not very precise, the increased error in the I_s/I_p determination is due to the mathematical relationship between measured and derived quantities. A still greater error would be expected in the determination of the effectiveness of lead screens if it were not for the fact that direct X-ray and lead-screen exposures were simultaneous and occupied adjacent film areas.

From other experiments[6] and the results given here, it is apparent that a large proportion of the secondary radiation has a greater average wave length than the primary but it is also certain that some of it is of the same wave length as the primary. It has been shown in a previous paper[7] that, as the thickness of the front lead foil screen is increased, the advantage of the additional thickness decreases rapidly. This may be *partially* due to filtration by the lead foil gradually making the primary and secondary radiations more nearly alike in quality. Except for the somewhat greater average path traversed by the scattered radiation in the lead, the lead screen could have no favorable effect on the radiograph as soon as the two radiations become similar in quality.

From a general knowledge of X-rays, it would be expected that radiographs of the same subject made at different kilovoltages might have different proportions of secondary radiation, that is, the amount of scattering per electron depends upon the wave length.[8] In this research, trials were made to determine the proportion of secondary radiation at different kilovoltages. For 5.1 cm. aluminum, exposures were made at 110, 130, 150, and 170 kv. constant potential. With 1.3 cm. steel and 0.078 cm. lead, exposures were made at 160, 180, and 200 kv. constant potential. No systematic difference was observed but, considering the lack of accuracy of this kind of work and the fact that any one voltage represents a large range of wave lengths, it is quite reasonable to expect that the wave lengths used at the different kilovoltages are so nearly alike, on the average, that no effect would be noticed. A wide range of kilovoltages was not convenient because of the great extremes of exposure times necessary. In actual radiography, the range of kilovoltages for a given thickness of material is limited for the same reason, so it seems that it is quite safe to say that there is no practical influence of the kilovoltage on the proportion of secondary radiation in the radiograph. Only a narrow range of kilovoltages is satisfactory for the penetration of a given subject, so the choice of radiographic technique is quite limited in this respect.

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[6] A. H. Compton and S. K. Allison, "X-rays in Theory and Experiment," D. Van Nostrand and Co., New York City (1935).

[7] Herman E. Seemann, "Some Physical and Radiographic Properties of Metallic Intensifying Screens," *Journal of Applied Physics*, Vol. 8, No. 12, December, 1937, p. 836.

[8] See for example:

John Read and Charles C. Lauritsen, "An Investigation of the Klein-Nishina Formula for X-ray Scattering in the Wavelength Region 50 to 20 X-Units," *Physical Review*, Vol. 45, No. 7, April 1, 1934, p. 433.

Trevor R. Cuykendall, "The Absorption of X-rays of Wavelength $50 \leq \lambda < 150$ X.U. by Elements of Low Atomic Number," *Physical Review*, Vol. 50, July 15, 1936, p.105.

Matthew T. Jones, "The Absorption of Ultra-Short X-rays by Elements of High Atomic Number," *Physical Review*, Vol. 50, July 15, 1936, p. 110.

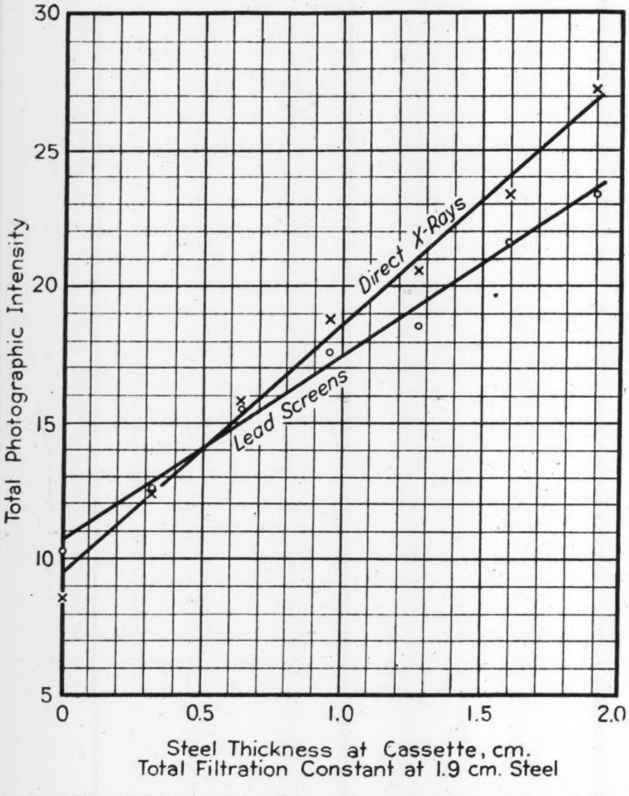

FIG. 6.—Evidence That Equal Layers of Steel Contribute Equal Amounts of Secondary Radiation to the Radiograph. (The two curves are plotted to the same scale for convenience and are not to be compared.)

The question arose in the course of this work as to what might be the distribution of the secondary radiation in the different layers of the specimen, or, what is the effect of absorption of the specimen on its own secondary radiation? To test this point, a specimen was made up of several plates of steel clamped together. An exposure was made and then one plate on the tube side was moved to a position near the tube. Another exposure was made and the next plate then moved near the tube. In this way, the intensity of the primary beam was maintained constant, being filtered by the same amount of material for all exposures, but secondary radiation was reduced by the removal of successive layers. The contribution of secondary radiation from the layers which were near the X-ray tube was known to be very small. The results of this experiment (Fig. 6) show that layers of equal thickness produce equal amounts of secondary radiation on the film. It would be expected, of course, that the layers on the tube side of the specimen would generate more secondary radiation since they absorb more of the primary beam than the layers near the film. The effect which compensates for this appears to be absorption by the layers near the film of the secondary radiation contributed by the layers near the tube. This straight-line relation, therefore, represents the partial compensation of scattering of one layer by absorption of its scattering in adjacent layers.

An experiment was performed in an attempt to correlate some of these results with more fundamental data on X-ray scattering. Two-hundred kilovolt X-rays were prefiltered with 0.4 cm. of copper to remove some of the softer components. The photographic intensity of this radiation was then recorded and the beam used in scattering measurements on 6.3 cm. aluminum, 1.3 cm. steel, and 0.052 cm. lead plates. These thicknesses gave comparable reductions in the intensity of the incident radiation and an approximate effective wavelength of 0.1 Å. The scatter per electron, presumably the same for different elements for the same wave length, was found to be 6.5×10^{-26} for aluminum, 2.7×10^{-26} for steel, and 150×10^{-26} for lead. These values are so far apart that it must be concluded that the several unknown factors, such as absorption of scatter by the specimen, angular distribution of scatter, proportion of modified wave length in the Compton effect, etc., are so different in these materials under radiographic conditions that not even approximate agreement in the scattering per electron is to be expected. It is probable that considerable X-ray fluorescence from lead is being recorded but not from aluminum or steel.

CONCLUSION

The paper is an outline of the principles of measurement and results of experiments performed to determine the amount of secondary radiation in the radiography of common industrial materials. Thicknesses were limited to those which can be conveniently radiographed without fluorescent intensifying screens. The principal conclusions to be drawn are: (1) that the proportion of secondary radiation from aluminum and steel increases almost linearly with thickness; (2) that lead screens may remove nearly one-half of the secondary radiation recorded when no screens are used; and (3) that no important reduction in secondary radiation is possible by adjustment of the kilovoltage within the range it is necessary to use for a given subject. These experiments are only an introduction to the study of this important problem in radiography. Further investigation is contemplated with the hope that future results may contribute not only to knowledge of the subject but to improvements in radiographic technique.

GAMMA-RAY RADIOGRAPHY[1]

By Gilbert E. Doan[2] and Shang-Shoa Young[2]

Synopsis

Certain similarities between radiographic exposures and solar eclipses are pointed out, and an equation based on these similarities is developed. This equation, if valid, would permit shorter source-to-film distances and substantially shorter exposure times than those now used. Experiments designed to test this equation confirm these expectations. The penumbral shadow plays a useful part in these short-range exposures.

In testing welds radiographically for internal flaws such as cracks, gas bubbles, slag, and lack of fusion, very sensitive methods must be used or these flaws will be missed because of their small size. Larger castings, on the contrary, usually have flaws of a type more readily detected. The present study undertakes to refine the accuracy and lower the cost of testing welds (and other objects of like importance) radiographically. The case considered is that of gamma rays but the principles seem applicable also to X-rays.

Part I—Theory

In present-day γ-ray radiographic testing, the most important free choice to be made in setting up the exposure is the choice of distance between radiant source and film, shown in Fig. 1 as d plus t. All the other values, such as the time of exposure and strength of source, are determined largely by the fixed conditions of the test involved. Heretofore, estimates based on experience have usually dictated the choice of the distance to be used. The optimum distance will, of course, be the one at which dangerous defects can be detected with certainty but with the shortest possible exposure time.

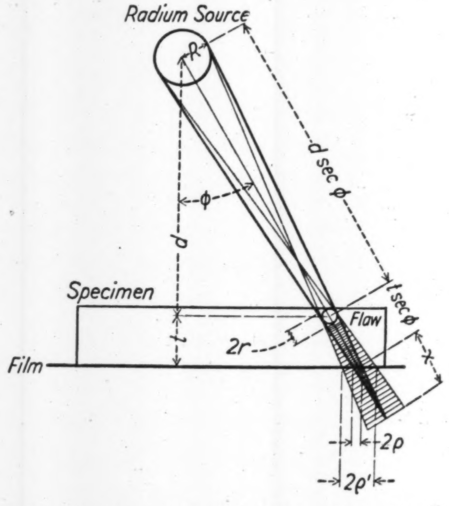

Fig. 1.—Geometrical Analysis of the Conditions in Radiographic Testing.

With this point of view in mind, a number of years ago one of the authors developed a formula by which it might be possible to calculate exactly the minimum distance permissible from radiant source to recording film in radiographic testing. Such a formula would promise sharp registry of all flaws of important size and at a minimum of exposure time.

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[1] Presented before the American Society for Testing Materials through the courtesy of the Fundamental Research Division of the Welding Research Committee of Engineering Foundation.

[2] Professor of Physical Metallurgy, and H. M. Byllesby Research Fellow, respectively, Lehigh University, Bethlehem, Pa.

Related to Fig. 1, the formula is as follows:

$$d = \frac{R - r}{r - \rho \cos \phi} \cdot t^{*} \ldots\ldots (1)$$

The purpose of these experiments is to test that equation. Figure 1 shows diagrammatically a radiant source of radius R, placed at a distance d from a specimen containing a flaw of radius r, the flaw lying at distance t from the film. The "shadow" of the flaw at the film is of radius ρ. In the general case the flaw will of course not lie in the shortest line between source and film, but in another line at some angle ϕ to the shortest one, as shown in Fig. 1.

To calculate the exact distance at which to place the radium, it would appear that we need only to decide what is the smallest diameter of flaw which is to be detected and to establish the minimum diameter of "shadow" $2\,\rho$ which can be observed on the film. This diameter of the smallest observable shadow was chosen arbitrarily as $\frac{1}{32}$ in. for purposes of the present experiments.

The calculation of distance d should of course be made for the most *unfavorable* condition possible, that is, for the flaw at the object surface farthest from the film (t will then be equal to the thickness of the object) and for an angle ϕ equal to zero. At this angle the flaw should cast its smallest but sharpest "shadow" on the film. It should be noted here that this paper deals primarily with *diameters* of flaws and shadows, measured at right angles to the rays, and not with flaw *depths*, although the latter aspect has been dealt with in earlier studies.

To illustrate the use of the formula, Table I gives the results of a typical calculation based on the following set of assumed conditions: The diameter of the flaw is assumed equal to the minimum thickness of flaw detectable by γ-rays, as determined by Norton and Ziegler in their tests of limiting sensitivity.[3] The radius of source R is assumed as 0.05 in. The smallest ob-

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[*] This formula can be derived from the geometrical relations shown in Fig. 1 with no more mathematics than fundamental algebra and trigonometry. For the convenence of the reader, the derivation is given below:

$$\frac{\rho \cos \phi}{x} = \frac{r}{x + t \sec \phi}$$

$$= \frac{R}{x + t \sec \phi + d \sec \phi} \text{ approximately;}$$

$$(x + t \sec \phi) \cdot \rho \cos \phi = rx;$$

whence

$$(x + t \sec \phi + d \sec \phi) \cdot \rho \cos \phi = Rx,$$

or

$$2) \quad t + d = \left(\frac{R}{\rho} - \cos \phi\right) x = \left(\frac{R - \rho \cos \phi}{\rho}\right) x.$$

Again by (1)

whence

$$t = \left(\frac{r}{\rho} - \cos \phi\right) x,$$

or

$$(3) \qquad x = \frac{t \cdot \rho}{r - \rho \cos \phi}.$$

Now, by (2) and (3)

$$t + d = t \frac{(R - \rho \cos \phi)}{(r - \rho \cos \phi)},$$

whence

$$d = \frac{(R - \rho \cos \phi - r + \rho \cos \phi)}{r - \rho \cos \phi} \cdot t$$

or

$$(4) \qquad d = \frac{R - r}{r - \rho \cos \phi} \cdot t.$$

For $\phi = 0$

$$(5) \qquad d_0 = \frac{R - r}{r - \rho} \cdot t \text{ and } d_0 > d$$

For a given d_0 $\quad r - \rho = (R - r) \frac{t}{d_0}$

or

$$(6) \qquad \rho = r - (R - r) \frac{t}{d_0}$$

[3] J. T. Norton and A. Ziegler, "Sensitivity of the Gamma-Ray Method," *Transactions*, Am. Soc. Steel Treating, Vol. 22, p. 271 (1933).

servable shadow is assumed as $\frac{1}{32}$ in. (ρ = 0.015 in.). The distance from flaw to film t is assumed equal to the thickness of the specimen; that is, the flaw is assumed to lie at the surface of the specimen farthest from the film. The calculated values of d plus t under these assumed conditions are shown in the last column. It will be noted that these distances are shorter than those commonly used. The calculations would therefore indicate that shorter times of exposure than those now used, with resulting economy both of time and radium expense are possible if Eq. 1 is valid. These exploratory calculations, based on Norton's limits, are given here only as illustrations of the possibilities presented in the use of the formula. In order to get *dense* and easily measurable shadows in the experiments necessary to test the formula with precision, deeper flaws were used than those calculated above, attention being concentrated on the *diameters* of the flaws and of their shadows, rather than on their depths, as pointed out above.

TABLE I.—RESULTS OF A TYPICAL CALCULATION.

R = 0.05 in.

Object Thickness t Same as Distance Film-to-Flaw t, in.	Flaw Diameters, $2r$, in.	Shadow Diameter on Film, 2ρ, in.	Optical Angle, ϕ, deg.	$\rho \cos \phi$ (for calculation), 0.015 × 1	Distance Source-to-Film, $d + t$, in.
1.0......	0.04 (4% of t)	0.03	0	0.015	6.00 + 1.0 = 7.00
2.0......	0.04 (2% of t)	0.03	0	0.015	12.00 + 2.0 = 14.00
3.0......	0.042 (1.4% of t)	0.03	0	0.015	14.50 + 3.0 = 17.50
4.0......	0.052 (1.3% of t)	0.03	0	0.015	8.73 + 4.0 = 12.73
5.0......	0.065 (1.3% of t)	0.03	0	0.015	5.00 + 5.0 = 10.00

Figure 1 and the formula show that the distance d would be especially important when the radiant source is larger in diameter than the flaw. When, on the contrary, the flaw is larger than the radiant source, the case is quite simple, as shown in Fig. 2. The principal precaution necessary when the flaw is larger than the source is simply to use distance d great enough to keep the shadow inside the edges of the film. When the shadow 2ρ is smaller than $\frac{1}{32}$ in. (the assumed minimum size of shadow observable on the film), d seems to become infinite; that is, the shadow would presumably not be observable at any value of d.**

If, however, the cone of partial shadow or penumbra, shown at $2\rho'$ outside the central cone of true (full) shadow in Fig. 1 could be depended upon to register a flaw, then it should be possible to shorten d still further and, using this partial shadow, still obtain a definite registry upon the film of any flaws within the object. The very short distance d involved in this proposal would be highly desirable, for instance in testing narrow cylinders with thick walls, where the radium must be placed at the axis of the cylinder and the films around the circumference. The radius of the penumbral shadow ρ' in Fig. 1 would be given by the equation

$$\rho' = r + (R + r)\frac{t}{d} \dots\dots (2)$$

It will be noted that the diameter of the penumbra increases as the distance d decreases.

As in the classical case of the solar eclipse, the penumbral shadow would

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** If the flaw, instead of being a drilled hole $\frac{1}{32}$ in. in diameter, were a crack $\frac{1}{32}$ in. wide and of some length, it might of course be readily observed on the film, whereas a single spot $\frac{1}{32}$ in. in diameter might be missed. The figure $\frac{1}{32}$ in. used in these tests is thus a pure assumption of limiting size.

not be expected to have a constant density, but beginning at its inner edge with a blackness equal to that of the true shadow, it should fade gradually until at its outermost edge it merged in density with the blank film. These features should appear also in radiographic penumbra.[4]

The experiments undertaken were designed to test both features: (1) the permissible shortening of d to obtain a true shadow of the flaw, and (2) the possibility of depending on the penumbral shadow at even shorter distances.

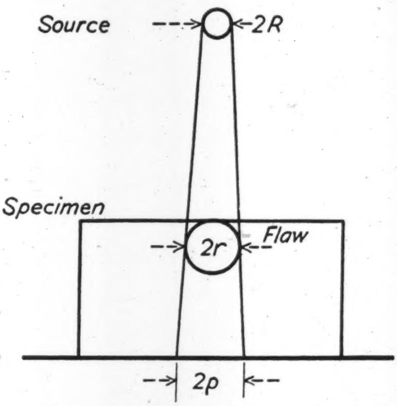

FIG. 2.—The Conditions Are Relatively Simple When the Flaw Is Larger Than The Radiant Source.

PART II—EXPERIMENT

Preparation of Samples:

The samples were prepared by making holes and slots of various sizes from $\frac{1}{64}$ in. up to $\frac{1}{4}$ in. in diameter in smoothly ground, mild steel blocks. Different specimen thicknesses were obtained by interposing blocks of various gage between these holes and the film. Care was taken that the thickness of the flaw with respect to the total thickness of each specimen was sufficient to produce a dense "shadow" such as would permit of accurate measurement. One of the specimens is shown in Fig. 3. Specimen thicknesses from $1\frac{1}{4}$ to $4\frac{1}{4}$ in. were used.

Other Materials:

The source of γ-ray was radium emanation of some 50 millicurie strength sealed in a glass bulb of spherical shape which, in turn, was put into a cylindrical brass capsule of 1-mm. wall thickness. Eastman Ultra-speed Dupli-tized X-ray films were chosen, and two films were interposed between three lead foil screens for each radiograph. A $\frac{1}{8}$-in. lead plate gave stiffness to the film mounting and protected the back of the film from scattered radiation. Developing and fixing powder used were those usually employed for X-ray films.

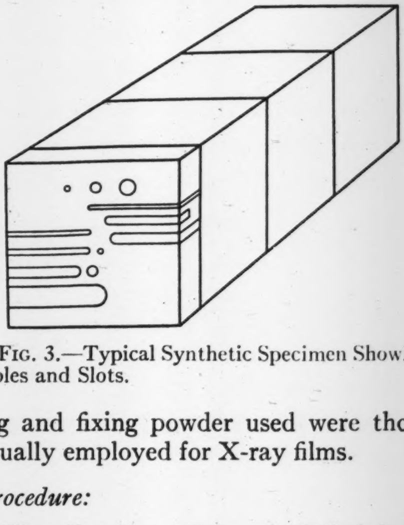

FIG. 3.—Typical Synthetic Specimen Showing Holes and Slots.

Procedure:

The distances chosen included both those calculated according to Eq. 1 and a range extending beyond these distances. In the latter cases the size of the "shadow" was computed from the distance d by the formula:

$$\rho = r - (R - r)\frac{t}{d} \ldots\ldots (3)$$

This is easily obtained by transposing Eq. 1 and solving for ρ while angle $\phi = 0$. The exposure time was cal-

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[4] For a fuller discussion of the shadow condition see Appendix I, p. 301.

TABLE II.—OBSERVED SHADOW DIAMETERS COMPARED WITH CALCULATED VALUES OF BOTH TRUE AND PENUMBRAL SHADOW DIAMETERS.

$R = 0.0689$ in.

Key				$\frac{1}{8}$-in. Holes and Slots			$\frac{3}{32}$-in. Holes and Slots			$\frac{1}{16}$-in. Holes and Slots			$\frac{3}{64}$-in. Holes and Slots			$\frac{1}{32}$-in. Holes and Slots			$\frac{1}{64}$-in. Holes and Slots		
Data	Films	t, in.	d, in.	2ρ Calculated, in.	Observed Shadow Diameters, in.	$2\rho'$ Calculated, in.	2ρ Calculated, in.	Observed Shadow Diameters, in.	$2\rho'$ Calculated, in.	2ρ Calculated, in.	Observed Shadow Diameters, in.	$2\rho'$ Calculated, in.	2ρ Calculated, in.	Observed Shadow Diameters, in.	$2\rho'$ Calculated, in.	2ρ Calculated, in.	Observed Shadow Diameters, in,	$2\rho'$ Calculated, in.	2ρ Calculated, in.	Observed Shadow Diameters, in.	$2\rho'$ Calculated, in.
T - 27	A - 12	$2\frac{1}{4}$	2	0.110	0.25	0.421	0.044	0.19	0.354	−0.022	0.13	0.298	−0.056	∞	0.255	−0.089	∞	0.221	−0.122	0	0.188
T - 5	A - 16	$2\frac{1}{4}$	6	0.120	0.19	0.224	0.077	0.13	0.171	0.034	0.09	0.138	0.013	0.06	0.115	−0.009	0.05	0.095	−0.030	0	0.073
T - 13	A - 15	$2\frac{1}{4}$	10	0.122	0.14	0.184	0.084	0.11	0.146	0.045	0.08	0.108	0.027	0.06	0.089	0.007	0.05	0.069	−0.012	0	0.050
T - 15	A - 14	$2\frac{1}{4}$	14	0.123	0.13	0.167	0.087	0.09	0.131	0.050	0.06	0.095	0.032	0.05	0.077	0.014	0.03	0.058	−0.004	0	0.040
T - 19	A - 13	$2\frac{1}{4}$	18	0.123	0.13	0.158	0.088	0.09	0.123	0.053	0.06	0.088	0.035	0.05	0.070	0.018	0.03	0.052	0.000	0	0.035
T - 17	A - 12	$2\frac{1}{4}$	22	0.124	0.13	0.152	0.089	0.09	0.118	0.055	0.06	0.085	0.037	0.05	0.066	0.020	0.03	0.048	0.003	0	0.031
T - 8	A - 11	$2\frac{1}{4}$	26	0.124	0.13	0.148	0.090	0.09	0.114	0.056	0.06	0.080	0.039	0.05	0.063	0.022	0.03	0.046	0.005	0	0.029
T - 28	A - 1	$4\frac{1}{4}$	0.57	0.031	∞	2.085	......	......	......	......	......	......	......	......	......	......	......	......	......	......	......
T - 29	A - 1	$4\frac{1}{4}$	2	0.099	0.38	0.684	......	......	......	......	......	......	......	......	......	......	......	......	......	......	......
T - 6	A - 7	$4\frac{1}{4}$	9.89	......	......	......	......	......	......	0.031	0.062	0.148	......	......	......	......	......	......	......	......	......
T - 3	A - 8	3	2.07	0.106	0.28	0.506	0.031	0.19	0.429	......	......	......	......	......	......	......	......	......	......	......	......
T - 22	A - 2	3	4	......	......	......	0.061	0.16	0.268	0.006	0.09	0.213	−0.023	0.06	0.186	......	......	......	......	......	......
T - 20	A - 8	3	5	......	......	......	0.068	0.19	0.233	......	......	......	−0.008	0.06	0.158	......	......	......	......	......	......
T - 26	A - 2	3	6	......	......	......	0.072	0.14	0.210	0.025	0.08	0.163	0.000	0.06	0.139	......	......	......	......	......	......
T - 30	A - 5	$3\frac{1}{4}$	1	......	......	......	−0.049	0.50	0.844	......	......	......	......	......	......	......	......	......	......	......	......
T - 16	A - 17	$3\frac{1}{4}$	4.86	......	......	......	0.064	0.20	0.249	......	......	......	−0.014	0.06	0.170	......	......	......	......	......	......
T - 12	A - 5	$3\frac{1}{4}$	10	......	......	......	0.079	0.16	0.169	......	......	......	......	......	......	......	......	......	......	......	......
T - 11	A - 3	$1\frac{1}{4}$	4.07	......	......	......	0.080	0.15	0.165	0.039	0.05	0.124	......	......	......	......	......	......	......	......	......
T - 7	A - 10	$1\frac{1}{4}$	6.77	......	......	......	0.086	0.12	0.137	......	......	......	0.031	0.06	0.081	......	......	......	......	......	......

NOTE.—The values of 2ρ are calculated by the equation, $\rho = r - (R - r)\frac{t}{d}$ where $R = 0.0689$ in. The values of $2\rho'$ are calculated by the equation, $\rho' = r + (R + r)\frac{t}{d}$ which can be easily derived from Fig. 1 by taking $\phi = 0$. ∞ and 0 indicate that no shadow is observed. Negative values indicate that the true shadows fall inside the specimen and should not be registered.

TABLE III.—OBSERVED SHADOW DIAMETERS COMPARED WITH THE CALCULATED VALUES OF BOTH TRUE AND PENUMBRAL SHADOW DIAMETERS.

$R = 0.125$ in.

Key				$\frac{1}{4}$-in. Holes and Slots			$\frac{7}{32}$-in. Holes and Slots			$\frac{3}{16}$-in. Holes and Slots			$\frac{5}{32}$-in. Holes and Slots			$\frac{1}{8}$-in Holes and Slots			$\frac{3}{32}$-in. Holes and Slots			$\frac{1}{16}$-in. Holes and Slots			$\frac{1}{32}$-in. Holes and Slots		
Data	Films	t, in.	d, in.	2ρ Calculated, in.	Observed Shadow Diameters, in.	$2\rho'$ Calculated, in.	2ρ Calculated, in.	Observed Shadow Diameters, in.	$2\rho'$ Calculated, in.	2ρ Calculated, in.	Observed Shadow Diameters, in.	$2\rho'$ Calculated, in.	2ρ Calculated, in.	Observed Shadow Diameters, in.	$2\rho'$ Calculated, in.	2ρ Calculated, in.	Observed Shadow Diameters, in.	$2\rho'$ Calculated, in.	2ρ Calculated, in.	Observed Shadow Diameters, in.	$2\rho'$ Calculated, in.	2ρ Calculated, in.	Observed Shadow Diameters, in.	$2\rho'$ Calculated, in.	2ρ Calculated, in.	Observed Shadow Diameters, in.	$2\rho'$ Calculated, in.
T - 9	B - 2	$3\frac{1}{4}$	1	0.250	∞	1.875																					
T - 15	B - 2	$3\frac{1}{4}$	$3\frac{1}{4}$	0.250	0.50	0.750																					
T - 18	B - 2	$3\frac{1}{4}$	6	0.250	0.38	0.521																					
T - 28	B - 2	$3\frac{1}{4}$	10	0.250	0.28	0.413																					
T - 7	B - 3	$3\frac{1}{4}$	$\frac{1}{2}$				0.018	∞	3.269																		
T - 40	B - 3	$3\frac{1}{4}$	3				0.188	0.50	0.727																		
T - 26	B - 3	$3\frac{1}{4}$	6				0.204	0.31	0.473																		
T - 34	B - 3	$3\frac{1}{4}$	10				0.210	0.25	0.371																		
T - 10	B - 4	$3\frac{1}{4}$	$1\frac{1}{4}$							0.026	∞	1.326															
T - 41	B - 4	$3\frac{1}{4}$	3							0.120	0.50	0.662															
T - 27	B - 4	$3\frac{1}{4}$	6							0.154	0.31	0.424															
T - 35	B - 4	$3\frac{1}{4}$	10							0.168	0.22	0.330															
T - 12	B - 5	$3\frac{1}{4}$	$2\frac{7}{16}$										0.031	∞	0.697												
T - 13	B - 5	$3\frac{1}{4}$	5										0.095	0.31	0.421												
T - 20	B - 5	$3\frac{1}{4}$	8										0.120	0.25	0.321												
T - 36	B - 5	$3\frac{1}{4}$	12										0.130	0.17	0.266												
T - 14	B - 6	$3\frac{1}{4}$	$4\frac{1}{4}$													0.031	0.25	0.412									
T - 19	B - 6	$3\frac{1}{4}$	8													0.074	0.19	0.278									
T - 29	B - 6	$3\frac{1}{4}$	12													0.091	0.16	0.227									
T - 17	B - 7	$3\frac{3}{8}$	$7\frac{11}{16}$																0.031	0.19	0.234						
T - 5	B - 7	$3\frac{3}{8}$	10																0.045	0.12	0.202						
T - 23	B - 7	$3\frac{3}{8}$	12																0.053	0.12	0.184						
T - 3	B - 8	$3\frac{1}{4}$	18																			0.031	0.06	0.119			
T - 2	B - 12	$3\frac{1}{4}$	$18\frac{3}{4}$																						−0.006	0.03	0.080
T - 48, 49, 50	B - 2, 6, 7	$1\frac{1}{4}$	6	0.250	0.31	0.354										0.099	0.12	0.203	0.065	0.12	0.165						
T - 43, 44, 45	B - 2, 6, 7	$1\frac{1}{4}$	20	0.250	0.25	0.281										0.117	0.12	0.148	0.085	0.09	0.115						
T - 11	B - 9	$3\frac{1}{4}$	2	0.250	0.75	1.06																					
T - 16	B - 9	$3\frac{1}{4}$	5	0.250	0.38	0.575																					
T - 21	B - 9	$3\frac{1}{4}$	8	0.250	0.31	0.453																					
T - 37	B - 9	$3\frac{1}{4}$	12	0.250	0.27	0.385																					
T - 30	B - 14	$3\frac{1}{4}$	18	0.250	0.25	0.340																					

NOTE.—The values of 2ρ are calculated by the equation $\rho = r - (R - r)\frac{t}{d}$, the same as in Table II, except that the radius of radiant source is, $R = 0.125$ in. here. The values of $2\rho'$ are calculated by the equation $\rho' = r + (R + r)\frac{t}{d}$. ∞ indicates that the shadow is diffuse and cannot be measure culated from the curves by C. S. Barrett, R. F. Mehl and R. A. Gezelius,[5] and correction for decay of the radium emanation was also made according to the table by the same authors. Overexposure of 50 per cent was allowed to obtain good contrast.

The chief difference between the first and the second experiment is that a radon bulb 0.1378 in. in inside diameter was used in the first, and a bulb of 0.250 in. in the second test. The larger bulb was used to increase the range of permissible flaw diameter. These bulbs were specially blown for the investigation. During the exposure the bulb, with its capsule, was set on a glass stand which is adjusted to point to the flaw in the shortest line. A typical arrangement of specimens for exposure is illustrated in Fig. 4. The size of the "shadow" on the film was measured visually by means of a scale. The calculated and the observed values of shadow diameter 2 ρ were tabulated side by side in Tables II and III.

Results:

Table II represents the results from the first and Table III those from the second experiment.

It will be noted that the shadow diameters *observed* in these experiments *diminish* from a greatly diffused, unmeasurable size at short distances of *d* down to the true dimension of the flaw as the distance *d* increases. This result must be that of penumbral registry, as illustrated in Fig. 1 and described above. For true shadows the diameters would *increase* as *d* increases, reaching the same diameter as the flaw when *d* is infinity. The true shadow, of course, lies inside the penumbral one but the gradual merging of the two shadows where they meet makes it impossible to separate one from the other in measuring diameters. All of the measurements

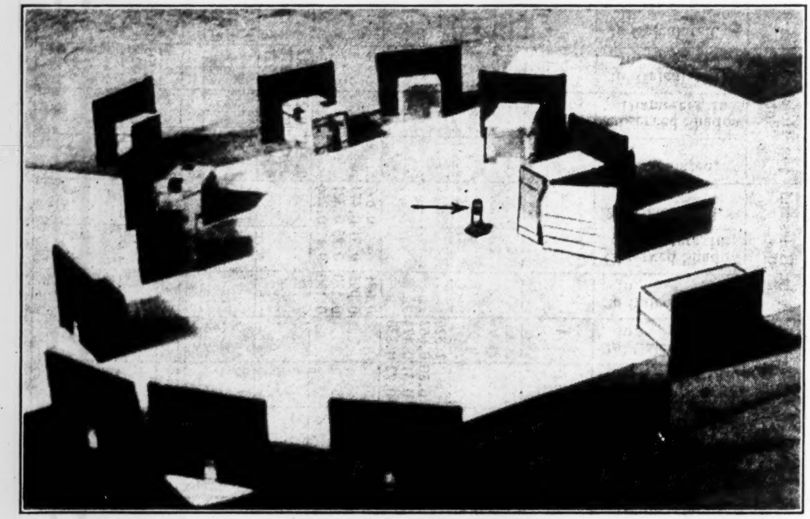

FIG. 4.—Typical Arrangement of Specimens for Exposure; Arrow Points to Radiant Source.

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[5] C. S. Barrett, R. F. Mehl and R. A. Gezelius, "The Technique of Radiography by Gamma Rays," *Metals and Alloys*, Vol. 1, December, 1930, p. 872.

G. E. Doan, "Gamma-Ray Radiographic Testing," *Journal*, Franklin Institute, Vol. 216, No. 2, August, 1933, pp. 183–216; No. 3, September 1933, pp. 315–385.

therefore apply to the outside or penumbral shadows.

The calculated penumbral values $2\rho'$ are also included in the tables. The agreement between these and the observed shadow diameters is good. It will be noted, however, that the observed shadow diameters, although tending in the right size direction for penumbra, are always smaller than the calculated penumbra diameters. This smaller observed shadow must be expected because of the above described fading out of the penumbra below an observable density at its outer edges. Since the shadows change in the order expected for penumbra, that is, they get smaller as the distance *d* increases, the penumbral theory accounts both for the decrease in shadow diameter as *d* increases, and for the slightly smaller penumbral diameters measured.[6] Even densitometer measurements of the films kindly made for us by the Eastman Kodak Co. do not show sharp transitions at the edges of the penumbra.

Furthermore, it is important to note that the registration of the penumbra *alone* actually is adequate to indicate a flaw, even when no true umbra or full shadow is obtained. In our experiment, in the column for $\frac{1}{32}$ in. in Table III, the calculated size of the "shadow" is negative; that is, there should be no umbra recorded. If the true shadow only were depended on, then the flaw would not be revealed on the film. But a "shadow" sufficiently dense for measurement was observed. This shadow can be no other than the penumbral shadow of the flaw. Penumbral registry is also shown in several columns in Table II, where actually observed "shadows" are recorded alongside negative values calculated on the old basis of true shadow only. By modifying the formula to yield the size $2\rho'$ of the penumbra, it may therefore be used to calculate approximate minimum distances *d* for any exposure.[7]

Limits of Shortening Distance d:

It should be noticed also that, in the second experiment as tabulated in Table III, with a source of radius $R = 0.125$ in., within a 3-in. distance from the source, where an umbra or full shadow should be registered according to calculation, either no "shadow" is formed or it is so diffuse that measurement could not be made. This is shown in the columns for $\frac{1}{4}$, $\frac{7}{32}$, $\frac{3}{16}$, and $\frac{5}{32}$ in. of Table III. The calculated value means that a full shadow is anticipated; the ∞ alongside it, however, indicates that neither the full shadow nor a penumbral one is observed. This absence of an observable shadow happened also in the first experiment, where a source of radius $R = 0.0689$ in. was used, as shown in the column for $\frac{1}{8}$ in. in Table II (although the distance *d* at which no shadow is obtained is shorter in this case where the smaller source is used, namely, within 1 in.). The limiting condition for *d* in the formula, below which no shadow is obtained, seems therefore to depend upon the size of the source and that of the flaw, upon their distance apart, and the distance from flaw to film. In the cases tested here, no shadow was obtained when the vertex angle between source and flaw was greater than 10 deg., as shown in Fig. 5. This generalization is only approximate, of course, because it takes no account of the distance from flaw to film. When the flaw is next to the film, it probably would register even at a much greater vertex angle than 10 deg.

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[6] See also Appendix II, p. 302.

[7] It will of course be understood that these considerations probably apply likewise to X-ray radiography where *R* becomes the radius of the radiant area on the anticathode of the X-ray tube, although assumptions based on another wave length do not always hold true. Attention is called especially to the paper by H. H. Lester, "X-ray Examination of Steel Castings," *Chemical and Metallurgical Engineering*, Vol. 28, January 7, 1923 p. 263.

As the specimen moves away from the source, the penumbral haze outside the true shadow becomes less and less noticeable. The successive sharpening of the shadow is best illustrated by a series of radiographs in Fig. 6 with distance indicated under each. Fully sharp shadow edges were obtained at 14-in. source-to-flaw distance when the bulb is 0.1378 in. in diameter, and at 20 in. when the bulb is 0.25 in. in diameter. The general condition for *sharp* pictures is found to be that in which the source and flaw subtend a common vertex angle of about 2 deg., as shown in Fig. 7. At smaller angles no increase in sharpness could be noted. At this distance the diameter of the penumbra approaches that of the true shadow, thus eliminating haziness of outline.

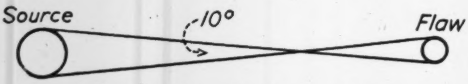


FIG. 5.—Conditions Under Which Flaw Registry Becomes Undependable.

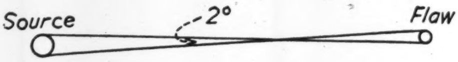
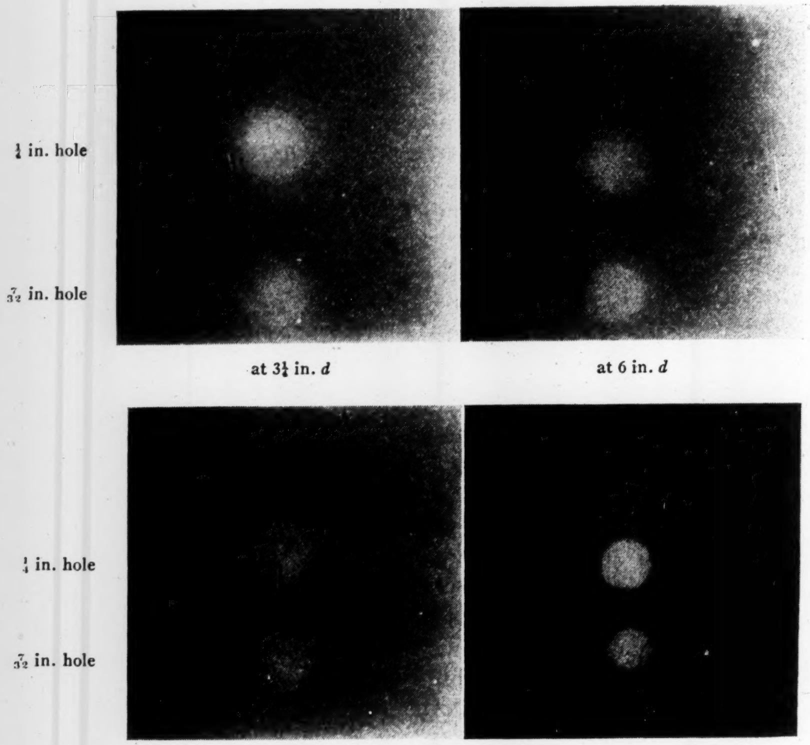


FIG. 7.—Conditions Under Which Shadow Edges Become Sharp.



at 3½ in. *d* at 6 in. *d*

at 10 in. *d* at 20 in. *d*

FIG. 6.—Disappearance of Penumbral Haze as *d* Is Increased.

Since the $\frac{1}{64}$-in. flaws did not register as shown in Table II even at a distance of 26 in. from the sources, further investigation of this matter of the lower limits of flaw diameter has been initiated.

SUMMARY

1. The results indicate that in gamma-ray radiography, dependable registry may be obtained from the penumbral shadow.
2. The formula developed for penumbral shadows makes possible a calculation of the minimum distance *d* permissible in obtaining certain registry.
3. At very short distances, such as those which form a vertex angle of 10 deg. or more between source and flaw, registry by penumbral shadow becomes undependable, unless the flaw lies very close to the film.
4. Even though a true shadow is registered at these short distances, it cannot be distinguished from the penumbral shadow because of the gradual shading of one into the other.
5. The diameter of the penumbral shadow always measures less than that calculated, because of the fading at its edges below an observable density.
6. Sharp "shadows" were obtained at 14-in. source-to-flaw distance with a $\frac{1}{8}$-in. radon bulb, and at 20 in. with a $\frac{1}{4}$-in. bulb. In these cases the source and flaw subtend a common vertex angle of about 2 deg. This result indicates again the superiority of a small bulb in gamma-ray testing.

Acknowledgments:

The assistance of Joseph B. Reynolds Professor of Mathematics, of the Radium Chemical Co., of the Eastman Kodak Laboratories and of Bradley Stoughton Dean of Engineering are acknowledged with thanks.

APPENDIX I

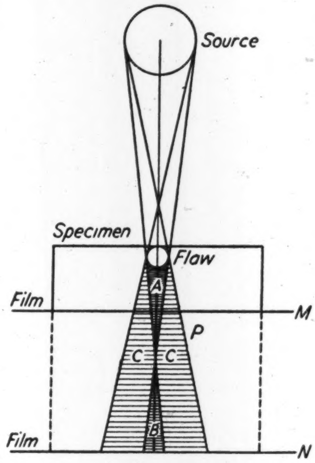


FIG. 8.—Penumbral Shadow at *c-c*.

Figure 8 shows fully the penumbral shadow at *c-c*. Beyond the tip of the true shadow *A* lies the "negative" shadow *B*. The intensity of shadow at any point, for example the point *P*, within *c-c* or *B* may be determined by drawing straight lines from the point in question tangent to the edges of the flaw. Thus for the point *P* the flaw intercepts rays from the source equal to only a part of the flaw area, the amount of interception depending on the location of the point *P* within the region *c-c*. Points within *B*, on the contrary, always suffer an interception of rays equal to the full area of the flaw.

The region *B* is therefore a region of constant shadow density, whereas in *c-c* the density varies steadily from its boundary at *B* to zero at its outermost edge. The position of the film as determined by the specimen thickness, together with the size of the flaw and of the source, and the distance from source to flaw will therefore determine which shadows are registered on the film. For thick specimens and short source-to-flaw distances the true shadow, being convergent, is lost to the film, whereas the penumbral shadow, including the negative shadow, being divergent, may be depended upon to register on the film.

APPENDIX II

To show the shadow sizes graphically, Fig. 9 is drawn. It shows that as d of Fig. 1 is increased all of the shadow diameters approach the true flaw diameter. The observed shadow diameter lies between the true and the calculated penumbral shadow diameters. The observed diameter is always larger, due to the penumbral rim. It is less than the calculated penumbral diameter, due to fading of the penumbra near its edges.

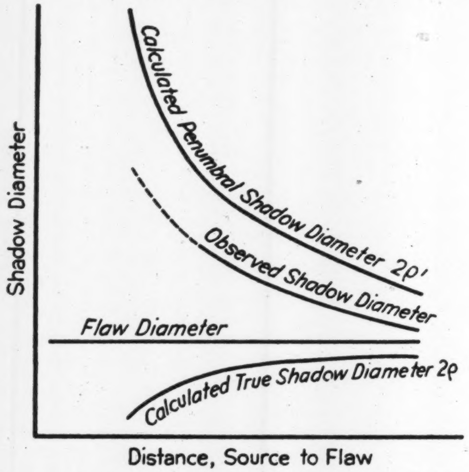


FIG. 9.—Showing Shadow Sizes Graphically.

A STUDY OF INTENSIFYING SCREENS FOR GAMMA-RAY RADIOGRAPHY

By Charles W. Briggs[1] and Roy A. Gezelius[2]

Synopsis

A number of different types of calcium tungstate intensifying screens were exposed to gamma rays and from the resulting film densities exposure charts were prepared. It was found that, when compared to the standard lead-foil intensifying screens, the calcium tungstate screens reduce the exposure time considerably. However, the intensity does not vary according to the inverse square law and the sensitivity or definition cannot equal that which may be obtained with the lead-foil screens.

It has been known for many years that a reduction in exposure time in gamma-ray radiography can be obtained if materials which fluoresce upon exposure to the rays are kept in intimate contact with the film during the exposure. Calcium tungstate has been found to be the most efficient intensifier for both X-rays and gamma rays. However, tests made at the time the original studies on gamma-ray technique were conducted indicated that the calcium tungstate screens available at that time did not possess enough advantages, when compared to lead screens, to warrant their use.

Since that time there has been considerable improvement in the calcium tungstate screens available for radiography. It was, therefore, deemed advisable to study several commercial screens in an attempt to improve the technique now used in gamma-ray radiography.

Six different sets of intensifying screens were obtained for the work. Sets C, D, E, and F were the ordinary commercial screens. Sets A and B were "experimental" screens loaned to the Naval Research Laboratory for this series of tests. All of these screens, with the exception of one set, were rather inflexible.

Testing Methods

Unfortunately, this test work was spread over a period of several months and, while the technique employed was the same in each case, the same amounts of radium were not available for testing all of the screens. Sets A, B, C, and D were tested with 25, 274, and 500 mg. of radium, set E was tested with 25 and 300 mg. of radium, and set F with 25 and 100 mg. of radium.

In each case density - exposure time curves were obtained at short distances ($6\frac{1}{2}$ to 8 in.). This was accomplished by using a cylindrical lead jug, $8\frac{1}{2}$ in. in diameter and 8 in. deep, with a hole in the center $1\frac{1}{2}$ in. in diameter by 6 in. deep. The radium was placed in the bottom of the hole, and steel plates (4 by 4 in.) of varying thicknesses were built up so that exposure times could be obtained for thicknesses of ½ to 2 in. in increments of ½ in. The time-density curves were constructed by exposing films for different lengths of time and reading the resulting densities on a

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[1] Physical Metallurgist, U. S. Naval Research Laboratory, Washington, D. C.

[2] Metallurgist, Taylor-Wharton Iron and Steel Co., High Bridge, N. J.; formerly with the Naval Research Laboratory.

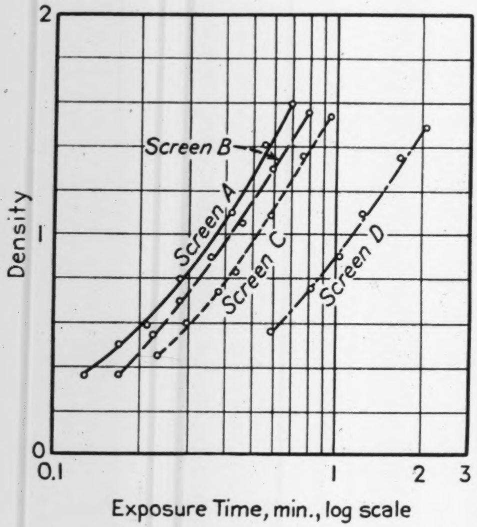

FIG. 1.—Time-Density Curves for 500 mg. of Radium at 7.5-in. Distance with 1½-in. Steel Plate.

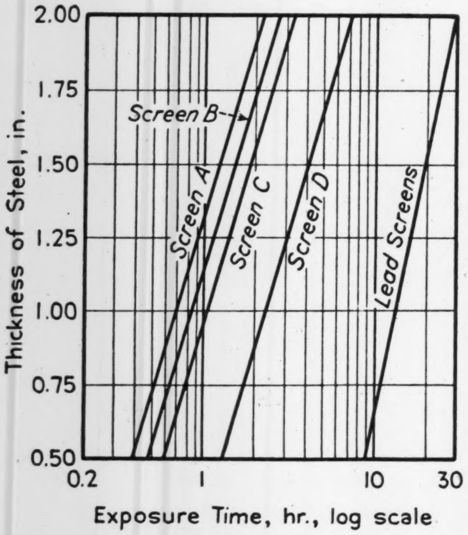

FIG. 2.—Exposure Curves for 25 mg. of Radium Calculated to a Film-to-Source Distance of 18 in., Density = 1.00.

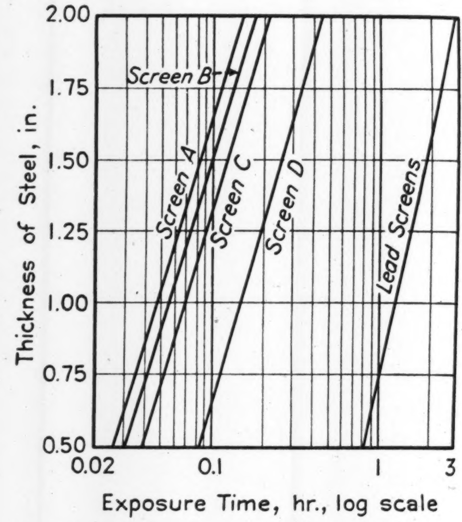

FIG. 3.—Exposure Curves for 274 mg. of Radium Calculated to a Film-to-Source Distance of 18 in., Density = 1.00.

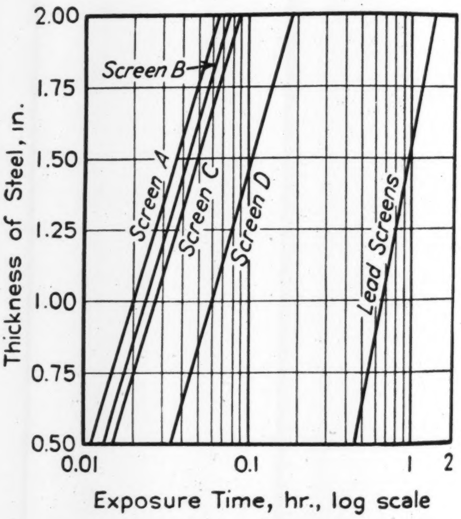

FIG. 4.—Exposure Curves for 500 mg. of Radium Calculated to a Film-to-Source Distance of 18 in., Screens Indicated.

Capstaff & Purdy densitometer. An example of one of these curves is shown in Fig. 1.

Previous work has indicated that a photographic density of 1.00 is preferable for gamma-ray radiography and time–thickness curves were constructed from the time–density curves using this density. The data obtained upon calculating the exposure required at a film-to-source distance of 18 in. are shown in Figs. 2, 3, and 4. On Fig. 5 a comparison is made of the exposure curves obtained with 25, 274 and 500 mg. of radium with screen D, all calculated to a common base of exposures at 18 in., with 100 mg. of radium. It may be noted on this chart that as the amount of radium is increased the exposure time per milligram decreased.

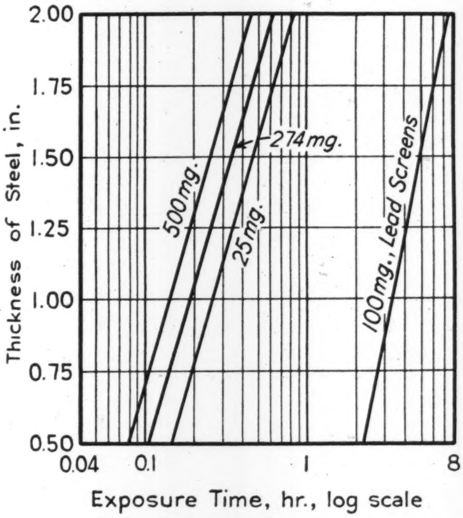

FIG. 5.—Comparison of Exposure Curves Obtained with 25, 274 and 500 mg. of Radium Calculated to a Common Base of Exposures at 18 in. and 100 mg. of Radium, Density = 1.00.

The most important shortcoming of the screens noted in these experiments was lack of definition and large grain in the film. This defect is undoubtedly due to secondary radiation which may be emitted by the specimen being radiographed or by the calcium tungstate screen itself. It is well known that the efficacy of any radiographic intensifying screen is actually due to the secondary radiation which is excited within the screen and reinforces the primary beam. The screen also tends to increase the definition by absorbing a portion of the scattered radiation emanating from the test piece. Therefore, on the second series of tests, the procedure was modified slightly in an attempt to control the secondary radiation and thus improve the appearance of the film. These modifications were:

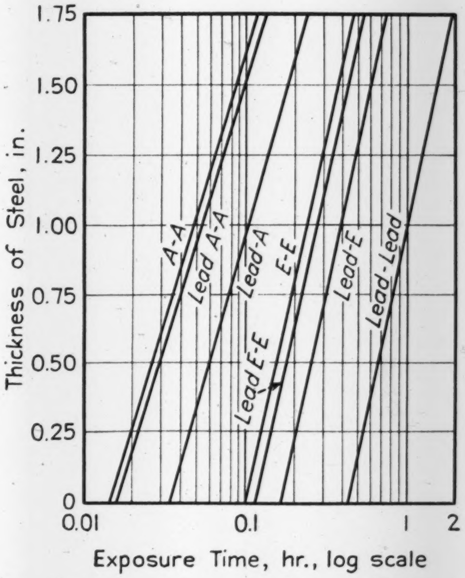

FIG. 6.—Exposure Curves for 300 mg. of Radium Calculated to a Film-to-Source of 18 in., Screens Indicated.

1. The use of lead foil (0.006 in. thick) behind the tungstate film nearest the source of gamma rays.
2. The replacement of the front screen with a lead screen.
3. The use of a slow, very fine grained

screen (screen D), alone and as in (1) and (2).

It has been found in previous work that radiographic film varies slightly and that unless the film used throughout the test bears the same emulsion number consistent results cannot be obtained. Therefore, the tests on screen A were repeated in order that a good comparison could be made. The combinations of screens used in this test were:

1. Screen A—film—screen A.
2. Lead—screen A—film—screen A.
3. Lead—film—screen A.
4. Screen E—film—screen E.
5. Lead—screen E—film—screen E.
6. Lead—film—screen E.
7. Lead—film—lead.

In each exposure in which a lead screen was used, the lead screen was nearest the radium. Heavy lead sheets were used in back of each cassette to screen out secondary radiation.

Exposure charts were prepared as before. Screens E exhibited the same characteristics as the calcium tungstate screens tested previously. The exposure curves obtained for 300 mg. of radium at a film-to-source distance of 18 in. are shown in Fig. 6.

The third series of tests were conducted with a screen that has recently appeared upon the market (screen F). As the thin extra lead screen (0.006 in.) did not appear to be of much value in eliminating the grain obtained in the film, the thickness of the filter was increased to $\frac{1}{32}$ in. The previous tests had indicated that the film-to-source distance was a factor in determining exposure times and a series of tests at various distances were therefore also included.

The outline of the procedure used is given below:

1. The 25-mg. capsule of radium was used as the source of gamma rays. Density - exposure time curves were obtained using short distances ($6\frac{1}{2}$ to 8 in.) on plate thicknesses from $\frac{1}{2}$ to 2 in. The data were collected on:
   - (*a*) Films exposed with a $\frac{1}{32}$-in. lead filter between the test piece and cassette and intensifying screens on both sides of the film. Normal X-ray development.
   - (*b*) Technique same as (*a*) except that films were developed twice the normal X-ray development time with potassium iodide in the developer.
   - (*c*) Technique same as (*a*) except that the $\frac{1}{32}$ in. lead filter was omitted.

2. The 25-mg. capsule of radium was used as the source of gamma rays. Density - exposure time curves at a distance of 18 in. were obtained on $\frac{1}{2}$ to $4\frac{1}{2}$-in. steel plates.

3. Item 2 was repeated at a 3-in. distance.

4. Item 2 was repeated using a 100-mg. capsule of radium as the gamma-ray source at distances of 3, 9, and 18 in.

Discussion of Results of Exposure Tests

The use of a $\frac{1}{32}$-in. lead filter between the specimen and the cassette increased the exposure time slightly. A film exposed without the lead filter had a density about 0.1 greater than a film exposed for an equivalent length of time with the filter. The grain size and the cloudiness in the finished film were reduced by the use of the filter.

Developing the exposed films for twice the normal time with potassium iodide in the developer decreased the exposure time required to obtain a density of 1.00. Overdevelopment had no effect upon the sensitivity or the general appearance of the film, even though an increase of approximately 0.3 in photographic density was brought about by overdevelopment. A comparison of the exposure times required for 25 mg. of radium at a film-to-source distance of 18 in. with normal development and overdevelopment is shown in Fig. 7.

Calcium-tungstate screens are fluorescent and appear to retain their fluoroscopic effect after removal of the primary beam. This has been indicated by an experiment in which four films were exposed to gamma-ray radiation at the same distance and for the same period of time. Two of these exposures were made with the films between 0.006 in. lead foil. The other two exposures were made using calcium-tungstate screens. One of the films exposed in contact with lead foil and one of the films exposed in contact with calcium-tungstate screens was removed from the cassette as soon as the exposure was completed and placed in a light-tight box. The other films, one exposed with lead foil and the other with calcium-tungstate screens were permitted to remain in contact with the screen used for a period of 72 hr. (The radium source had of course been removed.) At the expiration of the 72-hr. period the four films were developed simultaneously. The densities obtained on the films exposed with lead screens were practically identical. The density of the film exposed between calcium-tungstate screens and permitted to remain with them for 72 hr. was, however, considerably greater than that obtained on the film exposed between calcium-tungstate screens and then removed from the cassette as soon as the exposure was completed. It is evident, therefore, that the secondary radiation emitted by lead screens, principally beta rays, ceases as soon as the primary beam is removed, whereas the calcium-tungstate screens carry a latent image for some time after the removal of the primary beam. This retention of fluorescence tends to "build up" so that when long exposures are used the density is increased more than would be expected. The effect is illustrated in Fig. 8. The straight line is the exposure curve that would be expected if the "inverse square law" were the only consideration when transposing the data obtained at a distance of 7 in. The curved line is the exposure curve that

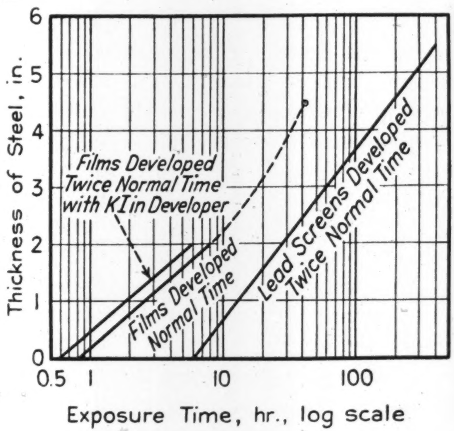

FIG. 7.—Exposure Curves for 25 mg. of Radium at 18 in. with Intensifying Screens F, and a $\frac{1}{32}$-in. Lead Filter, Density = 1.00.

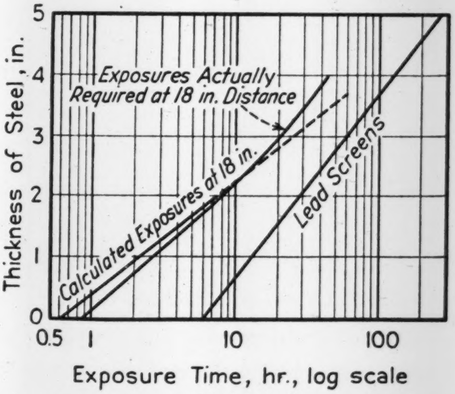

FIG. 8.—Exposure Curves for 25 mg. of Radium at 18 in. with Intensifying Screens F, and a $\frac{1}{32}$-in. Lead Filter, Normal Development, Density = 1.00.

was determined experimentally. It will be noted that the increase in distance has changed the slope of the curve and that in the upper portion of the curve the "fogging" due to the retained fluorescence tends to counteract the increase in exposure required for an increase in thickness.

Calcium-tungstate intensifying screens are also sensitive to the intensity of the source employed. That is, the exposure does not vary directly with the amount of radium used as is the case when lead screens are employed. When calcium-tungstate screens are used the exposure time *per milligram of radium* decreases as the intensity of the source increases. This phenomenon is illustrated in Figs. 5 and 9. In Fig. 9 the actual exposure curves for 25 and 100 mg. of radium are shown with the curve obtained when an exposure curve for 100 mg. is calculated from the experimental data obtained with 25 mg. of radium. It should be noted that the actual exposure times required with 100 mg. of radium are not only considerably less than the calculated values but that the slopes of the two curves are entirely different.

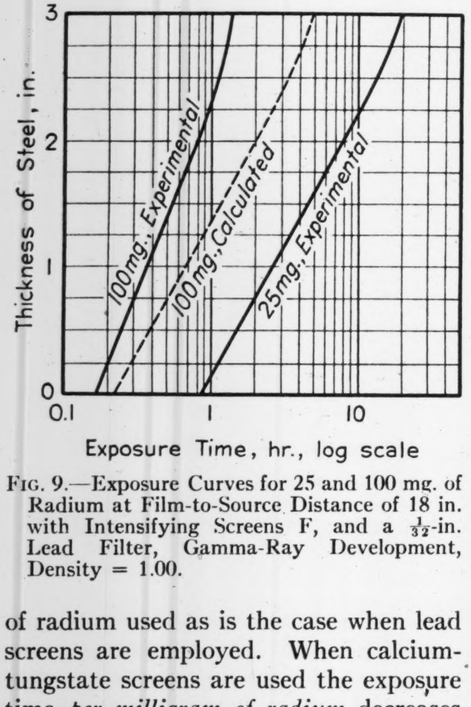

FIG. 9.—Exposure Curves for 25 and 100 mg. of Radium at Film-to-Source Distance of 18 in. with Intensifying Screens F, and a $\frac{1}{32}$-in. Lead Filter, Gamma-Ray Development, Density = 1.00.

If the exposure curves for 25 and 100 mg. of radium at distances of 3, 9, and 18 in. (shown in Fig. 10) are examined it may be seen that the slopes of the exposure curves vary with the film-to-source distance employed as well as with the intensity of the source. It should also be noted that exposure times are not given for thickesses greater than 1½ in. The curves were terminated at this thickness for, as may be seen in Fig. 7, at thicknesses greater than this fogging due to retained fluorescence becomes important. This fogging is dependent upon the actual time of exposure to some extent and therefore becomes more serious as the intensity of the source is decreased. Test exposures with 25 and 100 mg. of radium and test plates 1 and 3 in. thick indicate that at 3 in. even heavy lead markers are obliterated by fogging.

Time-distance exposure curves for 25 and 100 mg. of radium for several thicknesses are given in Fig. 11. It should be noted that the slopes of the curves are slightly different. The dotted line indicates what the slope of the curves should be to follow the "inverse square law." The increase in distance affects the intensification in much the same manner as a decrease in the intensity of the source.

It has been found that a similar condition exists in ordinary photography. Jones and Webb[3] in a review of "Reciprocity Law Failure in Photographic Exposures" point out that as early as 1892 Abney[4] found that when a candle was used for illuminating a photographic plate, and the illumination on the plate was decreased by increasing the distance between the candle and the plate, the photographic effect decreased more

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[3] L. A. Jones and J. H. Webb, *Journal*, Soc. Motion Picture Engrs., Vol. XXIII, No. 3, pp. 142-158 (1934).

[4] W. deW. Abney, *The Photographic Journal*, Vol. 18, pp. 254-260, 302-310 (1893-1894).

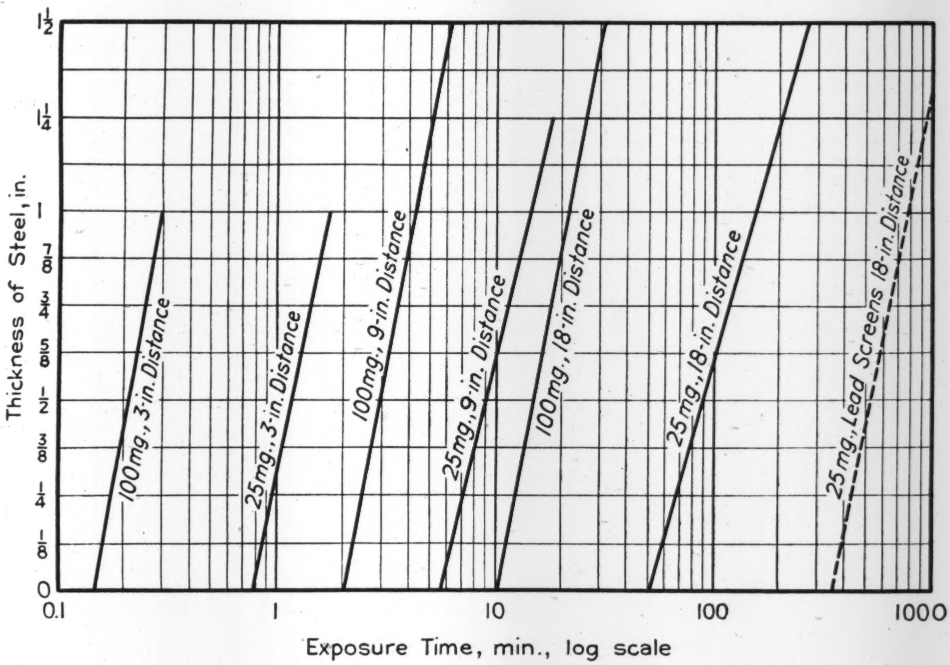


FIG. 10—Exposure Curves for 25 and 100 mg. of Radium with Intensifying Screens F, a $\frac{1}{32}$-in. Lead Filter, and Normal Gamma-Ray Development, Density = 1.00.

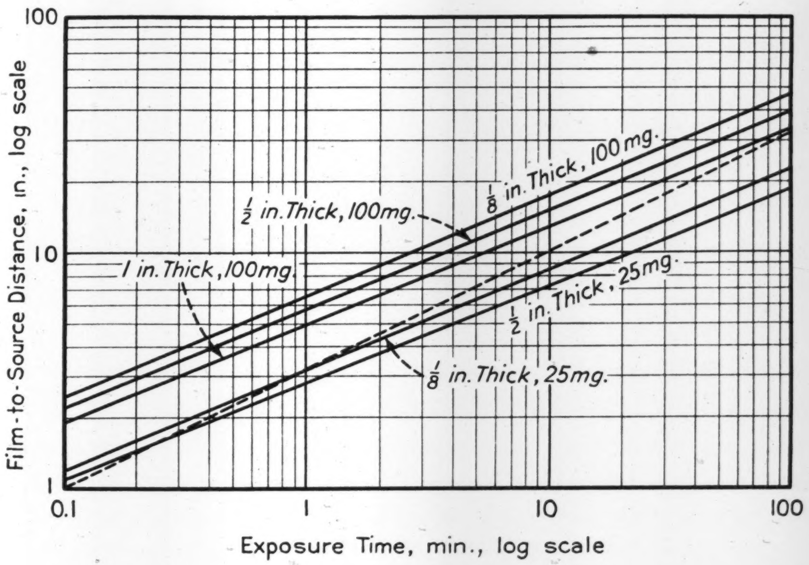
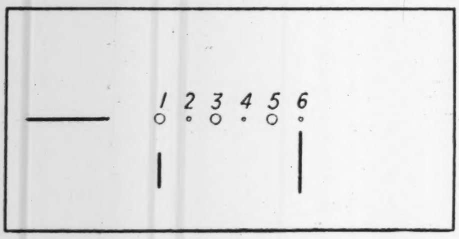


FIG. 11.—Time-Distance Exposure Curves for 25 and 100 mg. of Radium at Thicknesses Specified.

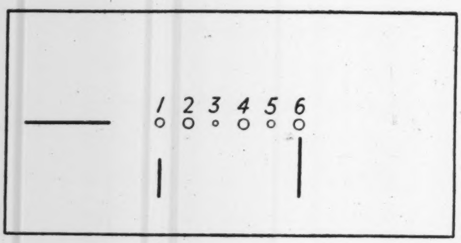
rapidly than would be expected. They also show that each type of emulsion has an "optimal intensity" at which the exposure required to obtain a definite density is less than that required at any other intensity.



1-in. Plate

Hole	Depth, In.	Diameter, In.	Depth, per cent of total thickness
No. 1	0.04	3/16	4
No. 2	0.03	0.03	3
No. 3	0.03	3/16	3
No. 4	0.02	0.02	2
No. 5	0.02	3/16	2
No. 6	0.04	0.04	4

3-in. Plate



Hole	Depth, In.	Diameter, In.	Depth, per cent of total thickness
No. 1	0.12	0.12	4
No. 2	0.06	3/16	2
No. 3	0.06	0.06	2
No. 4	0.09	3.16	3
No. 5	0.09	0.09	3
No. 6	0.12	3.16	4

FIG. 12.—Sensitivity Test Plates.

It is therefore possible that the deviation from the calculated exposure curves, obtained when calcium-tungstate screens are used with varying intensities at various distances, is due entirely to this phenomenon. It should be noted, however, that, when these screens are used with a constant distance and intensity, there still is a deviation from the exposure curve expected as the thickness is increased. This can be explained by the retention of fluorescence in the screens. The increase in thickness, of course, reduces the intensity of the rays reaching the photographic plate. It would be expected that, if the failure of the reciprocity law is similar to that found when the intensity of the source is decreased, the density would be decreased rather than increased. This coupled with the fact that the screens have been found to cause an increase in the density of the film, after the primary beam has been removed, leads one to believe that fluorescence is the cause of the deviation from the calculated curves when long exposures are used.

TABLE I.—SENSITIVITY OBTAINED BY THE USE OF INTENSIFYING SCREENS A, B, C, AND D.

	Sensitivity, per cent defect that can be seen	
	1-in. Plate	3-in. Plate
Lead Screen		
Defect position	1, 3, 5, 6	1, 2, 4, 5, 6
Defect, per cent of thickness	4, 3, 2, 4	4, 2, 3, 3, 4
Film characteristics	Fine grain, clear	Fine grain, clear
Screen A[a]		
Defect position	1, 3 faint	1, 4, 6
Defect, per cent of thickness	4, 3	4, 3, 4
Film characteristics	Large grain, cloudy	Medium grain, cloudy
Screen C		
Defect position	1, 3, 5	1, 4, 5, 6
Defect per cent of thickness	4, 3, 2 faint	4, 3, 3, 4
Film characteristics	Large grain, cloudy	Medium grain, cloudy
Screen D		
Defect position	1, 3 faint	1, 4, 6
Defect per cent of thickness	4, 3	4, 3, 4
Film characteristics	Large grain, cloudy	Large grain, cloudy

[a] Screen B is identical to screen A in sensitivity results.

It is interesting to note that a similar deviation has not been noted when lead screens are used, even though the exposures are considerably longer. The photographic effect is produced by beta rays when lead screens are used, whereas visible light is the principal factor when calcium-tungstate screens are employed. This may account for the different reactions obtained with the two types of screens.

TABLE II.—SENSITIVITY OBTAINED BY THE USE OF INTENSIFYING SCREENS A AND E WITH 0.006-IN. LEAD FILTER.

	Intensifying Screen	Sensitivity, per cent defect that can be seen	
		1-in. Plate	3-in. Plate
Lead-Film-Lead	L		
Defect position		1, 3, 5, 6	1, 2, 3, 4. 5, 6
Defect per cent thickness		4, 3, 2, 4	4, 2, 2, 3, 3, 4
Film characteristics		Fine grain, clear	Fine grain, clear
A-Film-A	A		
Defect position		1, 3 faint	1 faint, 4, 6
Defect per cent thickness		4, 3	4, 3, 4
Film characteristics		Large grain, cloudy	Medium grain, cloudy
Lead-A-Film-A	B		
Defect position		1	1, 4, 6
Defect per cent thickness		4	4, 3, 4
Film characteristics		Large grain, cloudy	Medium grain, cloudy
Lead-Film-A	C		
Defect position		1, 3	1, 2, 4, 5, 6
Defect per cent thickness		4, 3	4, 2, 3, 3, 4
Film characteristics		Large grain, cloudy	Medium grain, cloudy
E-Film-E	D		
Defect position		1, 3 faint	6
Defect per cent thickness		4, 3	4
Film characteristics		Large grain, cloudy	Medium-coarse grain, cloudy
Lead-E-Film-E	E		
Defect position		1, 3	1, 6
Defect per cent thickness		4, 3	4, 4
Film characteristics		Large grain, cloudy	Medium-coarse grain, cloudy
Lead-Film-E	F		
Defect position		1, 3	1, 6
Defect per cent thickness		4, 3	4, 4
Film characteristics		Large grain, cloudy	Medium-coarse grain, cloudy

SENSITIVITY TESTS

An attempt to determine the sensitivity of the various screens as compared to lead screens was carried out, using drilled test blocks. These test blocks contained 6 drilled holes distributed as shown in Fig. 12. Three of the holes in each plate were $\frac{3}{16}$ in. in diameter, and drilled to depths corresponding to 2, 3, and 4 per cent of the total thickness. The other three holes were drilled to corresponding depths, but in these cases the diameters of the holes were equal to the depths. The results obtained with the various screens are shown in Tables I to IV, inclusive. These tests were made at distances of 18 in. or greater, using 25 mg. of radium as a source unless stated otherwise in the table.

The results listed in Table I were made with screens A, B, C, and D without a filter. In each case the calcium-tungstate screens produced a cloudy effect upon the films which tended to obliterate the markings. If the exact positions of the drilled holes were not known it would be extremely difficult to determine a defect much less than 5 per cent of the plate thickness. The size of the drilled holes and the low contrast obtained precluded any hope of reproducing these films satisfactorily. Therefore the general appearance of the film has been noted in each case.

The results obtained by using a thin lead filter with screens A and E are given in Table II. It should be noted that the use of this thin filter, or the substitution of a lead screen for the front calcium-tungstate screen, has very little effect upon the sensitivity recorded and no effect upon the graininess of the finished film.

TABLE III.—DATA ON GAMMA-RAY EXPOSURES WITH INTENSIFYING SCREENS F.

Film	Plate Thickness, in.	Amount of Radium, mg.	Technique Film-to-Source Distance 18 in. Screens Indicated	Sensitivity Indicated by Penetrameter	
				Single Step Type	Step Type
No. 46	0.20	25	F	4.0 faintly	5.5 markedly
No. 47	0.20	100	F	4.0 faintly	5.5 markedly
No. 48	0.20	100	Lead	4.0 faintly	5.5 markedly
No. 49	0.20	25	Lead	4.0 faintly	5.5 markedly
No. 50	0.50	100	F	....	4.0
No. 51	0.50	25	F	....	2.2
No. 52	0.50	100	Lead	....	2.2
No. 53	0.50	25	Lead	....	2.2
No. 54	1.00	100	F	....	3.0
No. 55[a]	0.50	25	F	....	2.0

[a] Film-to-source distance 3 in.

A study of the sensitivity of screens F as compared to that obtained with the standard technique was also carried out at a film-to-source distance of 18 in. Most of this work was done on comparatively thin material as the screens appear to be more efficient on thin sections. The sensitivity was judged by placing a step penetrameter upon the specimen on the side nearest the source. This penetrameter had steps of 0.006, 0.011, 0.020, 0.030, and 0.041 in. in thickness with a $\frac{3}{16}$-in. diameter hole drilled in the center of each step. In addition to this, penetrameter strips with thicknesses of 0.003, 0.004, 0.006, and 0.008 in., containing a $\frac{3}{16}$-in. diameter drilled hole, were also placed on the specimen. The data obtained are given in Table III.

An examination of the data given in Table III indicates that on plates with a thickness of 0.20 in., the sensitivity is the same with lead or F screens. The films exposed with the F intensifying screens have poor contrast because of the cloudiness of the film. The definition is not as good as that obtained with lead screens.

TABLE IV.—COMPARISON OF SCREEN F AND LEAD SCREENS IN GAMMA-RAY PHOTOGRAPHY.

	Sensitivity	
	1-in. Plate	3-in. Plate
Screen F (100 mg. at 18 in.)		
Defect position	1, 3 faint	1, 4, 5, 6 all indistinct
Defect per cent of thickness	4, 3	4, 3, 3, 4 very foggy
Film characteristics	Very foggy and grainy	Very large grain
Screen F (100 mg. at 9 in.)		
Defect position	1, 3, 5 faint	
Defect per cent of thickness	4, 3, 2	
Film characteristics	Foggy	
Lead screen (100 mg. at 18 in.)		
Defect position	1, 3, 5, 6	1, 2, 4, 5, 6
Defect per cent of thickness	4, 3, 2, 4	4, 2, 3, 3, 4
Film characteristics	Fine grain, clear	Fine grain, clear
Lead screen (100 mg. at 9 in.) Same data as at 18 in.		

If the thickness of the specimen is increased to 1 in., the contrast becomes so poor, due to cloudiness and large grain, that it is extremely difficult to see even the outline of the penetrameter.

If the film-to-source distance is reduced to 3 in., the definition becomes very poor, and the shadow of the penetrameter is greatly enlarged. When this film was compared to that obtained with 0.006-in. lead screens it was found that the contrast is about the same but that the definition obtained with lead is better. It should also be noted that with the short film-to-source distance of 3 in., the density obtained on a 5 by 7-in. film changes rapidly, varying from a density of 1.4 at the center to 0.60 at the edge. Only the central portion of the film, a circle with a diameter of approximately 4 in., has a density suitable for proper interpretation.

Several tests were made on the 1- and 3-in. plates containing six drilled holes of various depths used to obtain the data presented above. These data are given in Table IV and when compared to the data given in Tables I, II, and III also indicate that as the thickness of the specimen increases the fogging and grain size increases.

In all radiographs made with intensifying screens of any type there is a tendency for the recorded image to appear slightly larger than the actual cavity being radiographed. The amount of "crawling" exhibited by a calcium-tungstate screen as compared to a lead screen was noted by passing gamma rays through a hole in a heavy lead cylinder and measuring the circle recorded on a film after the rays had penetrated 2 in. of steel. The primary image, that is, the well-defined dark circle, was $1\frac{11}{16}$ in. in diameter with both the calcium-tungstate screens and the lead screens. The circle defining the area of fogging around the primary image was $1\frac{7}{8}$ in. in diameter with lead screens, and 2 in. in diameter with the calcium-tungstate screens. The tendency to "crawl" is, therefore, slightly greater in calcium-tungstate screens than it is in lead screens.

CONCLUSIONS

With calcium-tungstate screens the exposure does not vary directly with the amount of radium used. The greater the amount of radium available the shorter is the exposure time per milligram. With lead screens the exposure varies directly with the amount of radium used.

Calcium-tungstate intensifying screens show considerable variations among themselves. Each screen studied had different characteristics.

Calcium-tungstate screens retain their fluoroscopic effect when long exposures are given, thereby acting to increase the film density.

The sensitivity recorded by using the calcium-tungstate screens was inferior to that of the lead-foil screens. Also the longer the exposure the poorer the definition due to the fogging or clouding over of the defects by the fluoroscopic effect retained by the screens.

As calcium-tungstate screens are limited to comparatively short gamma-ray exposures on thin sections, and the wave length of the gamma ray makes it unsuitable for use on thin sections, it appears that these screens are at the present time useful only in cases where good sensitivity and contrast are not required.

Acknowledgment:

The authors wish to acknowledge with appreciation the assistance of H. E. Seemann of the Kodak Research Laboratories for his constructive review of the manuscript.

DISCUSSION

MR. HERBERT R. ISENBURGER[1] (*presented in written form*).—The authors, Messrs. Briggs and Gezelius, must be admired for the tremendous amount of work they have done in an attempt to improve present day gamma-ray technique.

A little over four years ago the writer had the same idea and obtained as source of radiation a radon preparation which was used to exhaustion. Some 20 samples of various sizes, shapes and materials were exposed under various conditions using different screen materials. The negatives revealed nothing new except for one thing.

Among the objects under exposure, which ranged from 2-in. steel plates, 1-in. copper plates, 4-in. aluminum block to small zinc base die castings, were some welded fuselage structures. Some of these joints were exposed at great distance using intensifying screens. The result was a better image than with lead foil or without any screens. While we did not obtain as much definition and contrast as we did with X-rays and no screens, the result was remarkable and checks with the authors' statement that screens are more satisfactory on thinner materials.

The reason for this phenomenon may be the fact that in gamma-ray testing, we are using rays which are comparable in wave length to X-rays excited by over 1,000,000 v. Thus most of the gamma-radiation penetrates the screens and the secondary radiation is being absorbed by the screens which, in this case, act as filters, hence permitting a clearer shadow picture. Whether this has any practical value remains to be seen.

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THE EXAMINATION OF METALS IN POLARIZED LIGHT

By L. V. Foster[1] and J. E. Wilson[1]

Synopsis

The significance of illuminating and examining opaque surfaces in polarized light is discussed. We have shown that a specular reflecting surface may alter the brightness of a beam of light if the surface is tilted obliquely to the plane of vibration of the beam of illumination. This is the reason for scratches on highly polished surfaces showing bright in an otherwise extinct field when examined between crossed nicols. Most metal oxides are in nature colored but when polished and examined by means of vertical illumination reflect sufficient light to obscure their color completely. If examined between crossed nicols, the surface reflection is eliminated and their true color becomes visible.

A brief description of the way existing microscopes can be corrected to make observations in polarized light is given. A more extensive description of a new polarizing vertical illuminator which leads to more efficient bright field illumination is included. This illuminator, furthermore, provides an improvement in the quality of polarized light. The microscope in which this illuminator is incorporated has many novel features which aid in its use for both visual and photographic purposes, whether used in polarized light or bright-field applications.

Applications of polarized light to the identification of copper oxide, copper sulfide, chromic oxide and glassy silicates are illustrated, and the use of this technique in studies of grain size, preferred orientation and identification of phases in aluminum alloys are also illustrated. Other applications such as the measurement of anodic coating thickness on aluminum alloys are described. It is pointed out that only by systematic investigation can the full utility of the polarizing microscope be realized in any specific field of investigation.

Although polarized light has been a commonly used tool of the mineralogist and petrographer for many years, its application to the examination of metal microstructures is a recent development.

In the following discussion it is intended to summarize briefly the optical phenomena involved in the observation of metals under polarized light and to bring out by means of typical examples the method of utilizing these phenomena in metallographic investigations.

Nature of Polarized Light

Ordinary light consists of vibrations of a heterogeneous nature radiating from some source. The eye or a photographic plate or light-sensitive cell cannot distinguish between heterogeneous light vibrations and homogeneous vibrations. However, when ordinary light passes through transparent material or is reflected at the boundary surface of two different materials a change may take place in some of the directional vibrations of the ordinary light. When such a change takes place and the resulting transmitted or reflected vibrations become restricted so that they are no longer heterogeneous, the vibrations are said to be polarized. These vibrations may be plane polarized, vibrating in a plane, circularly polarized, vibrating in a circle, or elliptically polarized, vibrating in an ellipse. The eye will detect no difference between any of them and the heterogeneous vibrations. The only way they can be recognized is by using an optical device which will produce the same kind of vibrations as those just mentioned.

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[1] Optical Engineer, and Metallurgist, respectively, Bausch & Lomb Optical Co., Rochester, N. Y.

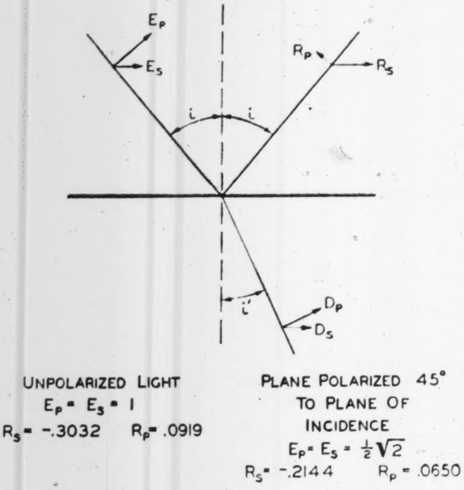

FIG. 1.—Ordinary Light Coming from the Left Is Incident on the Boundary Between Two Media.

The reflected light and transmitted light are partially polarized.

Take as a specific example, skylight reflected from the surface of a piece of glass. This light, to the eye, appears the same, except for intensity, as that of the sky itself. Hold between the eye and the piece of glass a polarizer and rotate it and it will be seen that in two orientations of the polarizer the skylight vanishes. The reason for this is that even though the original skylight was heterogeneous, the plate of glass reflects the light as plane polarized, and when the plane of polarization of the polarizer is perpendicular to the plane of polarization of the glass plate, no light will pass through the polarizer. Polarized light can be obtained by reflection from the surface of water, metals, lacquers, wood, paper and many other materials. The porportion of the reflected light which is polarized is a function of the angle of incidence of the light on the surface and the index of refraction on both sides of the boundary surface. Fresnel has expressed this in the following two equations:

$$R_s = -E_s \frac{\sin (i - i')}{\sin (i + i')},$$

and

$$R_p = E_p \frac{\tan (i - i')}{\tan (i + i')}$$

where R_s and R_p = the amplitudes of the reflected vibrations perpendicular to and parallel to the plane of incidence, respectively,

E_s and E_p = the electric forces perpendicular to and parallel to the plane of incidence, respectively, and

i and i' = the angles of incidence and refraction at the surface.

If light is incident at normal incidence (i = 0 deg.) there is no polarization. This assumes that $E_s = E_p$; then $R_s = R_p$. Referring to Fig. 1, if ordinary light ($E_s = E_p$) is incident at 45 deg. on a surface of $^{n}D = 1.5$, then

$R_s = -0.3032$ and $R_p = +0.0919$. This shows that the light is partially polarized. If the original light is polarized in the plane of incidence, then $E_p = 0$ and $R_s = -0.3032$ and $R_p = 0$. If the original light is polarized 45 deg. to the plane of incidence and its amplitude is 1, $E_s = E_p$. E_s and E_p can be found by resolving the amplitude into its two components (Fig. 2): $E_s = E_p = 1/2\sqrt{2}$. Then $R_s = -1/2\sqrt{2} \times 0.3032$ and $R_p = 1/2\sqrt{2} \times 0.0919$. The light is still plane polarized, but before reflection had an amplitude of $1/2\sqrt{2}$ in the two principal meridians and after reflection is oriented at $\tan^{-1} \dfrac{0.3032}{0.0919}$ or 73° 8′ away from the plane of incidence.

Effects of Transparent Media:

If we consider polarized light transmitted by an isotropic substance and examine the beam of transmitted light by means of a nicol prism, we can rotate the latter to a position where the beam of light is extinguished. If we rotate the isotropic substance, about any axis, the beam of light remains extinguished. That is, the isotropic substance has no effect on the beam of polarized light. If we substitute for the isotropic substance an anisotropic substance, the beam of light may reappear. Rotation of the anisotropic substance will either have no effect on the intensity of the beam or it will cause the beam to pass from extinction to maximum brightness four times during one complete revolution. In the former case, the anisotropic substance has been so located that its optical axis is coincident with the axis of the beam of light. In the latter case its axis is not coincident with the axis of the beam. The substance has made a change in the nature of the polarized light if brightness of the beam has been restored. This change may have been due to the production of circular or elliptical polarization, which was produced because of the birefringence of the material. It may have been produced because of a rotation of the plane of polarization. These are the only occurrences whereby brightness of the beam of light could be restored. The two causes can be distinguished from one another by means of compensators. That is a circular or elliptical vibration can be neutralized by adding between the polarizer and analyzer a suitable circular or elliptical vibration. Such compensators are known as a quartz wedge, Berek compensator, Babinet compensator, Johannsen compensator, Wright graduated quartz compensator, etc. If a compensator will not restore extinction, the plane of polarization has been rotated and a rotation of the analyzer can be made to restore extinction. In the case of circular or elliptical vibrations, the resulting brightness of the beam is not without color, which may be of the primary spectrum or the secondary or some higher order depending upon the birefringence and thickness of the material producing the vibration. Rotation of the plane of polarization will

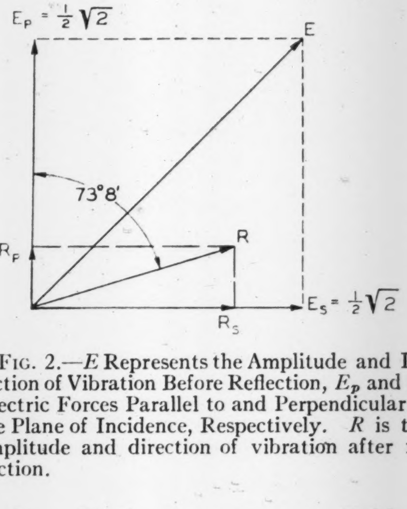

FIG. 2.—E Represents the Amplitude and Direction of Vibration Before Reflection, E_p and E_s Electric Forces Parallel to and Perpendicular to the Plane of Incidence, Respectively. R is the amplitude and direction of vibration after reflection.

also produce color due to rotary dispersion of the material, but this color cannot be compensated for by rotating the analyzer to restore extinction. It can only be restored when neutralized with a rotatory material of opposite sign and like dispersion.

Effects of Opaque Media:

If we project a beam of polarized light at normal incidence on an opaque surface such as a polished metal, and put an analyzer in the reflected beam, we should expect to be able to extinguish the beam of light by rotating the analyzer. When the analyzer is rotated to such a position, the analyzer and polarizer are said to be crossed. If the opaque surface is rotated about the axis of the beam of light, that is, about its normal, the beam of light is still extinguished. However, if the surface is rotated about an axis in itself and if this axis is not perpendicular to or parallel to the plane of polarization of the incident light the beam of light will not be extinguished. However, a rotation of the analyzer can be made which will restore extinction.

Reflection from Metal Surfaces:

When a beam of light falls on a metallic surface, part of it is reflected and part of it is absorbed or transmitted into the metal. Since it is not the purpose of this paper to explain the theory of this statement, only the necessary facts will be given (**1**).[2] The propagation of light in absorbing media involves equations of motion which are complex. The complex factor is the absorption. When a plane polarized light beam is obliquely incident on a metal surface, the reflected light beam is elliptically polarized (**2**). The constants of ellipticity can be measured and from them the coefficient of extinction and the refractive index of the metal determined. These values, computed by various observers, differ widely. The various workers have shown that a polished layer is either amorphous or consists of very small crystals and that the layer may be as thick as 500 Å which is of the order of the distance to which light penetrates (**3**). Metals have strong absorption bands which account for the red colors of copper and gold. Their absorption differs considerably and the result is that their reflecting power is different. For example, silver has a reflection of 95 per cent, steel 58 per cent, nickel 62 per cent, lead 62 per cent, and platinum 70 per cent. These values are of little use in the examination of metals, because these metals rarely occur together. It is to be noted, however, that this is one reason why some metals give a stronger beam of reflected light than others.

Metals may be isotropic or anisotropic. Ordinary vertical illumination permits one to recognize differences in light absorption. Examination in polarized light allows a qualitative or quantitative determination of their refractive index, their absorption and their double refraction. A discussion of the appearance of anisotropic crystals will be left for the latter part of this paper.

APPARATUS

The equipment which has been available to the metallurgist for the examination and photography of metal surfaces, has, until the last few years, been confined to the so-called bright field illumination.

To examine metal surfaces in polarized light it is necessary to place between the light source and the vertical illuminator a nicol prism. The nicol prism is a rhomb of calcite which has been cut in two pieces diagonally from one corner to the other and recemented together with balsam or linseed oil. The nature of the prism is such that when a beam of heterogeneous light is passed into it, the resulting transmitted beam is plane polarized. In order to make use of the illumination of the surface by polarized light it is necessary to place between the specimen and the eye an analyzer. The analyzer is a second nicol prism which may be placed over the eyepiece. The application of nicol prisms to the conventional metallurgical microscope has certain disadvantages which have been set forth by one of the authors in another paper (**4**). Within the last few years European manufacturers of metallurgical microscopes have built into their instruments a polarizer and tube analyzer, both being easily removable to allow ordinary bright field observations.

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[2] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 328.

Polarizing Vertical Illuminator:

A new optical device, known as a polarizing vertical illuminator, has been developed to study the surfaces of metals in polarized light. The two parts, *A* and *B*, of Fig. 3, are made from one piece of calcite with the optic axis parallel to the end of the prism and in the plane of the cemented surface. The two parts are cemented together with a cement having approximately the same index of refraction as the extraordinary ray. Light from the source travels along the axis of the microscope objective and, as in a nicol prism, is divided on entering the calcite into two rays, one the ordinary and the other the extraordinary. The ordinary is refracted and absorbed by black paint on the side of the prism while the extraordinary passes on through the prism, through the objective and to the specimen. The plane of vibration of this light which reaches the specimen lies in the plane of the drawing. The light illuminating the specimen is plane polarized and a perfect reflecting specimen would reflect the plane polarized light unaltered and it would pass back through the prism along its original path. The prism acts as a polarizer for light incident on the specimen and as an analyzer for light reflected from the specimen. It should be pointed out here that the light reflected at the cement calcite boundary follows the laws of refraction of the ordinary ray since this is the only ray which can be reflected. The index of calcite for the ordinary ray is 1.6585 and the index of the cement is 1.4864. Thus there can be no astigmatism along the path the light travels from the objective to the eyepiece. This is mentioned here because nicol prisms such as the Halle, or Glan Thompson transmit the extraordinary ray and produce astigmatism for all converging or diverging pencils of light.

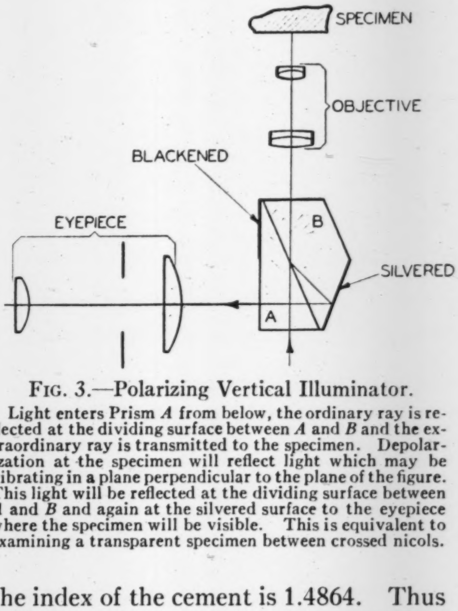


FIG. 3.—Polarizing Vertical Illuminator.

Light enters Prism *A* from below, the ordinary ray is reflected at the dividing surface between *A* and *B* and the extraordinary ray is transmitted to the specimen. Depolarization at the specimen will reflect light which may be vibrating in a plane perpendicular to the plane of the figure. This light will be reflected at the dividing surface between *A* and *B* and again at the silvered surface to the eyepiece where the specimen will be visible. This is equivalent to examining a transparent specimen between crossed nicols.

This prism illuminator can also be used to view metal surfaces in bright field illumination, that is, so that specular regions appear bright and grooves such as result from etchants appear black. Bright field illumination is secured by placing between the prism and the objective a quarter wave plate with its slow ray direction making an angle of 45 deg. with the vibration plane of the prism. The plane polarized light after emergence from the prism passes through the quarter wave plate and becomes circularly polarized. It is reflected by the specimen and again passes through the quarter wave plate. It has thus had a 90 deg. change of phase and is now vibrating perpendicular to the plane of the drawing. This light is fully reflected at the cement calcite layer to the silvered surface and to the eyepiece. The specimen appears in this kind of illumination exactly as it does when illuminated with the customary plane glass illuminator. It is to be observed, however, that of the initial intensity of light, 50 per cent is transmitted through the prism. There is about a 20 per cent loss by two transmissions through the quarter wave plate which makes the intensity of the light about 40 per cent of its initial quantity when it enters the prism after having been reflected by the specimen. All of this light will be reflected at the two reflecting surfaces to the eyepiece. Thus, this prism is about four and one-half times as efficient as the plane glass reflector.

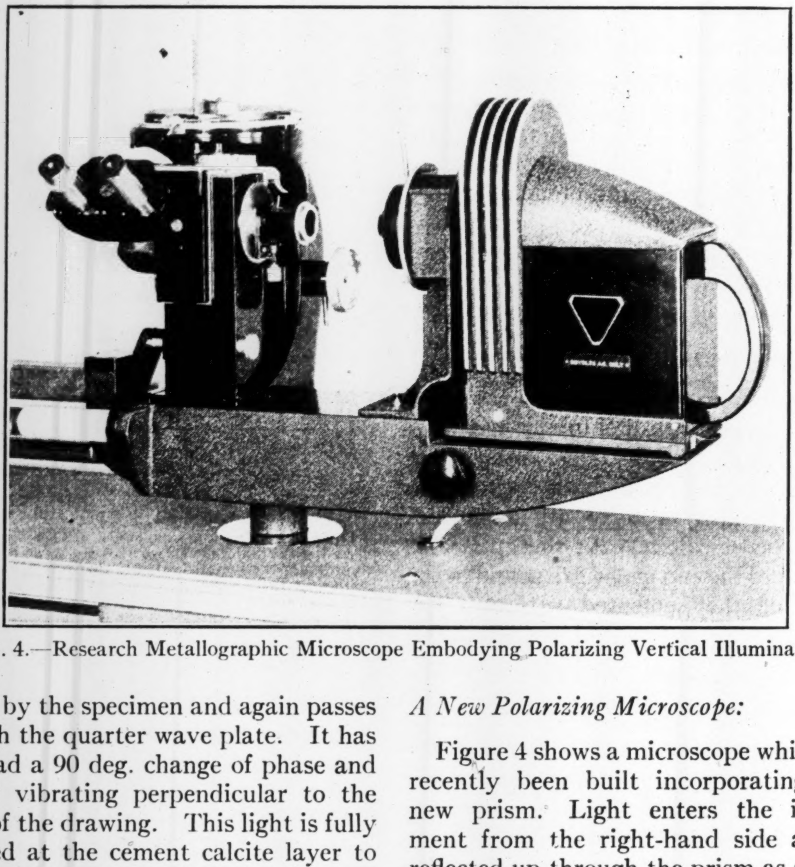
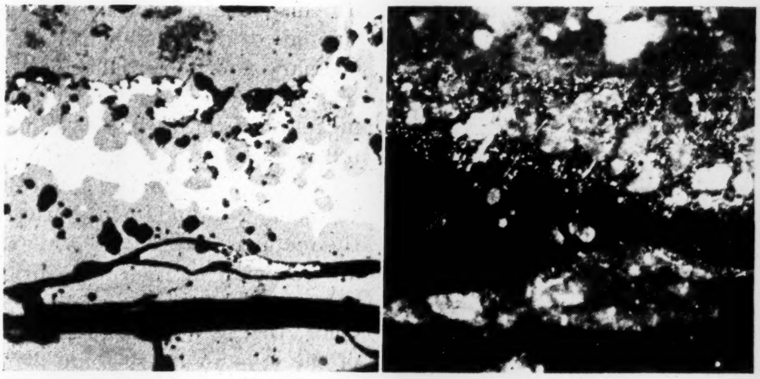


FIG. 4.—Research Metallographic Microscope Embodying Polarizing Vertical Illuminator.

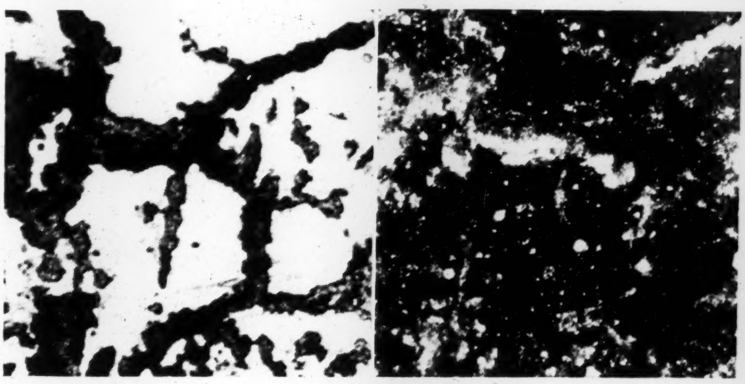
A New Polarizing Microscope:

Figure 4 shows a microscope which has recently been built incorporating this new prism. Light enters the instrument from the right-hand side and is reflected up through the prism as shown in Fig. 3. A rotating disk containing a clear aperture, a quarter wave plate, and a half wave plate is mounted above the prism. When the clear aperture is over the prism, the specimen is examined between crossed nicols. When the quarter wave plate is over the prism, the specimen is examined in bright field illumination and when the half wave plate is over the prism the specimen is examined in sensitive tint illumination. The stage of this microscope is completely revolving on ball bearings. On top of the rotating stage is a mechanical stage for moving the specimen in two directions at right angles to each other. These motions are each 1 in. long. In the tube through which the light enters the microscope are two diaphragms, one for diaphragming the aperture of the objective and the other for diaphragming the field. The aperture diaphragm may be displaced and rotated in order to give oblique illumination in any azimuth. The microscope is equipped for visual observation with a binocular body. By turning a lever on the back side of the microscope the light may be diverted from the visual eyepiece to the camera for making photomicrographs. A rod running the full length of the camera bed on the back side of the microscope provides coarse focusing adjustment and a similar rod running the full length of


(*a*) Ordinary light.

(*b*) Polarized light. Visually the gray to white constituent is red (copper oxide), the black is blue-violet (copper sulfide).

FIG. 5.—Mixture of Copper Oxide and Sulfide Scale on Copper. (White Constituent) (× 250). Unetched.


(*a*) Ordinary light.

(*b*) Polarized light. The light constituent is bright green.

FIG. 6.—Chrome Oxide in Nickel Chromium Alloy (× 250). Unetched.

the camera on the observing side provides remote control of the fine adjustment.

APPLICATIONS

Before discussing applications of equipment such as described above, it should be pointed out that polarized light is far more useful for visual microscopy than for photomicrography. All specimens which are anisotropic and have anisotropic crystals in them change their appearance radically when rotated. As has been pointed out earlier, if the specimen specularly reflects light without change in phase, it looks totally dark when examined in polarized light. If the specimen is anisotropic it will look dark in one orientation and bright in an orientation 45 deg. from the first, with various shades of brightness between the two. Some specimens, however, produce color effects in polarized light and the color effects cannot be photographed except by the use of color plates.

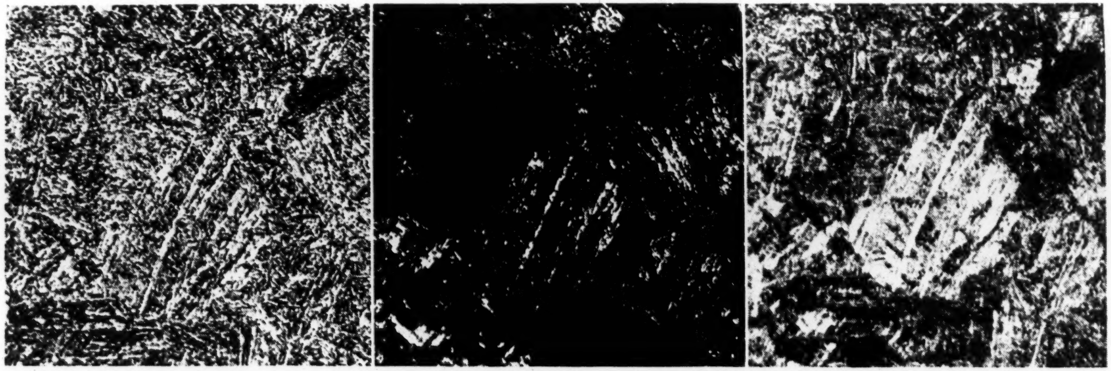
Use of Color Effects for Identification:

Many of the effects noted by visual examination in polarized light are difficult to reproduce in photomicrographs. The radical change in appearance of anisotropic substances on rotation of the microscope stage which gives rise to a change from extinction to brightness with every 45-deg. change in orientation, and the beautiful color effects produced in certain types of oxides and by the use of the sensitive tint plate are phenomena which can only be suggested by still pictures. Such color effects can often be used for identification purposes. For example, copper oxide and sulfide both appear blue-gray in ordinary illumination, as shown in Fig. 5 (*a*) (5). On changing to polarized light, the copper oxide appears a beautiful crimson while the sulfide retains the blue to purple color. In the black and white print, Fig. 5 (*b*), the crimson is shown as gray to white, the sulfide reproduces a dead black, while the narrow band of copper metal, white in Fig. 5 (*a*), is just a shade lighter than the sulfide. The practical value of the color distinction is obvious in this case. Another example of a colored oxide is shown in Fig. 6, in ordinary and polarized light. This is a section from a piece of a high chromium-nickel electric furnace element which failed in service. In Fig. 6 (*a*), the appearance is of gray intergranular corrosion product. In polarized light, the metal matrix is dark while inclusions are revealed in their true bright green color as shown in Fig. 6 (*b*). The green color, together with extinction of some of the crystals on rotation of the stage, identifies the corrosion product as chromic oxide, which crystallizes in the hexagonal system.

It should be emphasized that color effects of the type just illustrated are due to elimination of specular reflection, thus permitting the normal color of the substance to appear.

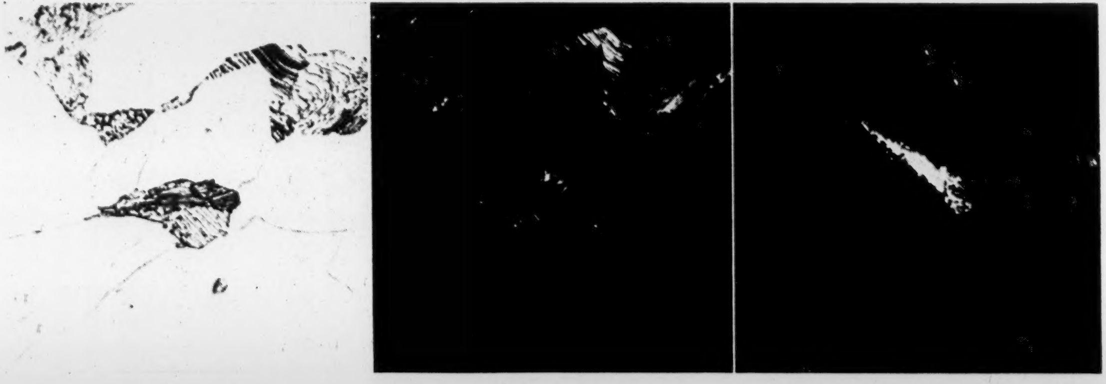
Grain Size:

The use of polarized light to investigate grain size of complex heat-resistant castings has been suggested by Ziegler and Haughwout (6). An example of its use to clarify the grain size and orientation of the forged structure of S.A.E. No. 1040 steel is given in Fig. 7. The grain pattern of the prior austenite which is observed with difficulty in bright field illumination, becomes quite definite in polarized light (Fig. 7 (*b*)) and by use of the sensitive tint plate, the grain contrast is further improved (Fig. 7 (*c*)). In general, low-carbon steel will show a pink matrix of ferrite, and with yellow to green pearlite grains with the sensitive tint plate inserted.



(*a*) Ordinary light. (*b*) Polarized light. (*c*) Sensitive tint plate. Colors are yellow to green on a pink background.

FIG. 7.—S.A.E. No. 1040 Steel (Forged Structure) (× 100). Etched in 3 per cent Nital.



(*a*) Ordinary light. (*b*) Polarized light. (*c*) Same as (*b*) except for 45 deg. rotation. Note change in the extinction of pearlite grains.

FIG. 8.—S.A.E. No. 1020 Steel, Cold Drawn (× 1000). Etched in 1 per cent Nital. 1.25 N. A.

Preferred Orientations:

Studies of cold-worked structure are facilitated by use of polarized light, since anisotropy of structure is set up by the severe plastic deformation. Figure 8 (*a*) is a longitudinal section of cold-drawn S.A.E. No. 1020 steel in bright field illumination. This same field in polarized light is shown in Fig. 8 (*b*).

It is noteworthy that parts of the pearlite grains are extinguished in this orientation while rotating the stage 45 deg. causes a reversal of extinction, as in Fig. 8 (*c*). Thus the lamellae must make a definite angle to the plane of polarization in order to reflect light which can pass through the analyzer. In the illustrated case, pearlite lamellae which have directions parallel to the bottom of the print are extinguished. This measure of orientation has been suggested by von Schwarz (7) as a means of controlling flow direction in such applications as cold heading of bolts.

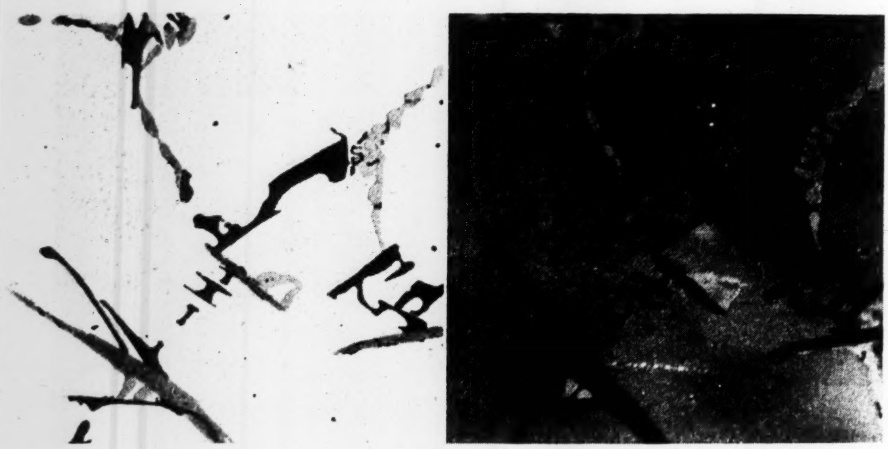
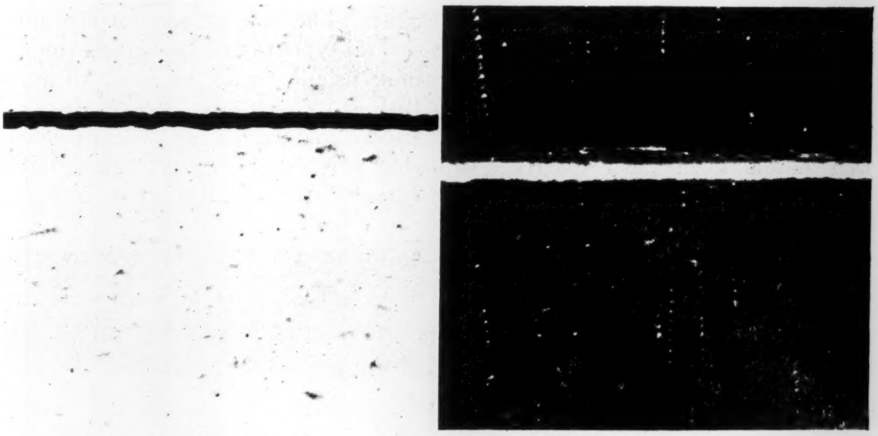

(*a*) Ordinary light. (*b*) Polarized light crossed nicols.

FIG. 9.—Microconstituents in Aluminum-Copper-Silicon-Iron Casting Alloy (× 500).

Aluminum Alloys:

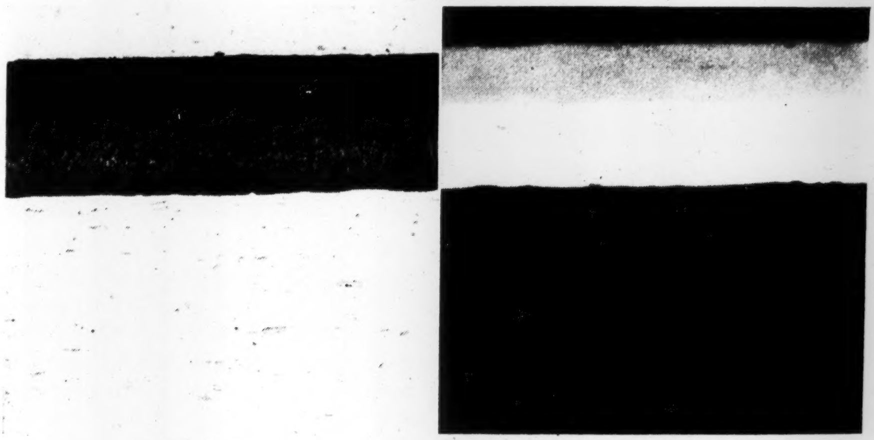
F. Keller of the Metallurgical Division of the Aluminum Research Laboratories at New Kensington, Pa., has found that polarized light is advantageous in the identification of microconstituents and inclusions. It can assist in the determination of the characteristics of oxide coatings produced by the anodic oxidation process and in the study of paint and other coatings. Quoting Mr. Keller:

"The microconstituents that occur in aluminum alloys consist of pure metals, solid solutions and intermetallic compounds. Many of these constituents, when examined by polarized light with crossed nicols, show polarization colors; each constituent develops a characteristic color which aids in identification. In consequence, it appears possible to work out a scheme for the identification of constituents in aluminum alloys by determining the polarization colors and the degree of rotation of the plane of polarization. As an illustration, the microconstituents in an aluminum-copper-silicon-iron casting alloy are shown in Fig. 9 (*a*) as they appear when examined under ordinary light. The same constituents are shown in Fig. 9 (*b*) as they appear when examined by polarized light under crossed nicols. The colors of these constituents aided in their identification.



(*a*) Ordinary light. (*b*) Polarized light, crossed nicols.

FIG. 10.—Anodic Coating Formed in Chromic Acid (× 500).



(*a*) Ordinary light. (*b*) Polarized light, crossed nicols.

FIG. 11.—Anodic Coatings Formed in Sulfuric and Oxalic Acid Electrolytes (× 500).

"The use of the polarized light has been found very helpful in the examination of oxide coatings produced by the anodic oxidation process on aluminum alloys. These coatings are usually produced in sulfuric, chromic or oxalic acid electrolytes. They consist of a surface coating which is from 0.00005 to 0.0001 in. in thickness. One of the best methods for determining thickness, continuity and other characteristics of these coatings is by the examination of polished cross-sections of the anodized product. The oxide coatings on aluminum, in general, do not reflect very much light back through the optical system of the microscope; consequently, they appear as narrow dark bands in a light matrix. Frequently, it is hard to distinguish these coatings from crevices between the individual specimens or from included polishing materials. When anodically coated specimens are examined by polarized light with crossed nicols, the anodic coatings are revealed as light colored bands in a dark matrix. In addition, anodic coatings made with different electrolytes have different optical characteristics. It is possible, therefore, to identify the anodic coatings with respect to the type of anodizing solution which was employed. Furthermore, it is possible to determine in some instances whether or not the coatings have been subjected to a sealing treatment and to determine the type of sealing treatment.

"The interpretation of the results of the microscopic examination of anodically coated samples of aluminum can be accomplished most satisfactorily by visual examination because the actual polarization colors can be compared. It is possible, on the other hand, to make a photographic record of these coatings which will be more satisfactory than a similar record made with ordinary light. The appearance, for example, of several types of anodic coatings on aluminum is illustrated in Figs. 10 and 11. In Fig. 10 (*a*) a cross-section through an anodic coating formed in a chromic acid electrolyte is shown as it appeared at a magnification of 500 when using ordinary light. The same coating is shown in Fig. 10 (*b*) as it appeared by polarized light with crossed nicols. Actually, this anodic coating had a pearly gray color. In Fig. 11 (*a*) coatings formed in sulfuric acid and oxalic acid electrolytes are shown adjacent to each other as they appeared when examined under ordinary light. It is just possible to determine that two different coatings are present. In Fig. 11 (*b*), however, the same coatings can be distinguished readily when examined by polarized light.

"Polarized light has been found of

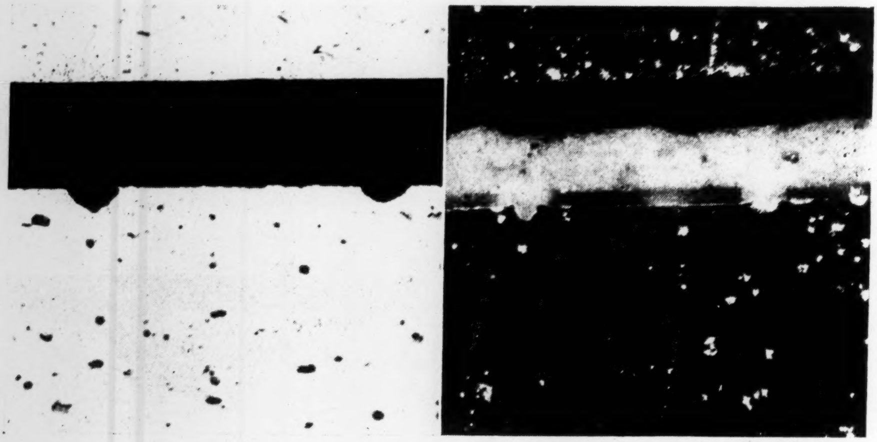

(*a*) Ordinary light. (*b*) Polarized light, crossed nicols.

FIG. 12.—Section Through an Anodized and Painted Aluminum Sheet (× 500).

advantage, also, in the examination of samples of aluminum which have been anodized and painted. Take, for example, the specimen illustrated in Fig. 12 (*a*). This specimen of sheet had been anodized and then painted with a priming coat and a clear lacquer coat. The micrograph taken with ordinary light does not reveal these separate coatings. With polarized light, however, the various coatings are revealed as shown in Fig. 12 (*b*) and can be identified by their polarization colors."

Photomicrographs reproduced as Figs. 9 to 12 inclusive, were not made on the instrument described in this paper but were made on instruments available at the Aluminum Research Laboratories.

Miscellaneous Applications:

An interesting effect of polarized light may be used to distinguish glassy silicates from other types of round nonmetallic inclusions. This is the so-called "optical cross" which is evident in several of the small round inclusions of Figs. 5 (*b*) and 6 (*b*). The cross is composed of two dark bands at right angles to each other produced because the reflected light from the sides of the spherical inclusions in planes parallel with the planes of vibration of the polarizer and analyzer undergoes no change of phase and consequently remains black, while in all other planes a rotation of the plane polarized light impinging on the sides of the spheres takes place and this light cannot be extinguished by the analyzer and thus leads to brightness. Consequently, when several of these inclusions are visible in the microscope field, the crosses will all have the same orientation.

Certain other inclusions such as tin oxide in bronzes, may be easily identified by the intense brightness produced in polarized light, due to the effects of anisotropy discussed above.

The study of alloy equilibria is facilitated in many cases by examination of specimens under polarized light. Many phases which appear as similar constituents under bright field illumination are distinguishable by different color effects or the exhibition of anisotropy with polarized light. Eash and Upthegrove (8) in studying the copper-rich copper-nickel-tin alloys found that the θ phase could be distinguished in this manner from the δ' phase.

The work of von Schwarz on nonferrous (5) and ferrous (7) applications gives a good survey of European literature, while the recent publication of Hoyt and Scheil (9) indicates the utility of the polarizing microscope in the identification of non-metallic inclusions in steel. In discussing the applicability of polarizing methods to metallography, the consensus of opinion has been that empirical methods based on comparison with known standard specimens offer the greatest possibilities at present, although the quantitative measure of the intensity of polarization effects has been considered in recent literature (10, 11).

Acknowledgments:

This paper has been in a sense a cooperative project and the cooperation of F. Keller in supply the aluminum data and L. L. Wyman, who sent us the samples of chromic oxide and copper oxide is especially appreciated. We also wish to acknowledge the assistance of J. V. Butterfield of the Scientific Bureau, Bausch & Lomb Optical Co., for making the Kodachrome slides.

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THE EFFECT OF USING A BLEND OF PORTLAND AND NATURAL CEMENT ON PHYSICAL PROPERTIES OF MORTAR AND CONCRETE

By W. F. Kellermann[1] and D. G. Runner[1]

Synopsis

This paper presents data obtained during a laboratory study of mortars and concretes made with portland cement and blends of portland and natural cements. The purpose of the investigation was to determine the effect of replacing a portion of the portland cement with natural cement on the physical properties of mortar and concrete.

Tests on mortars included crushing strength, flexural strength, absorption, density, durability and volume change. Two portland cements, differing considerably in chemical composition, and two sands of widely different mineral composition were used in this portion of the investigation. Tests on concrete specimens fabricated in the laboratory included crushing strength, flexural strength and modulus of elasticity, using the same two portland cements with local fine and coarse aggregate. One brand only of natural cement was used in all specimens fabricated in the laboratory.

In addition to tests on laboratory fabricated specimens, a series of tests was made on cores taken from four concrete pavements in New York State in which two different natural cements were used. In each of these roads portland cements conforming to four different classifications as to compound composition were used. Core specimens were taken from sections of each of these pavements in which straight portland cement was used as well as from sections in which blends of portland and natural cement in the proportion of 6:1 and 5:2 were used. These specimens were subjected to alternate freezing and thawing in water and in a 10 per cent solution of calcium chloride.

These tests indicated that, whereas strength was reduced by replacing a portion of the portland cement with natural cement, resistance to alternate freezing and thawing was, for certain combinations of materials, substantially increased.

Within the past few years considerable scaling on concrete pavements has occurred in the Northern States due to the use of chemical salts for ice removal. In an effort to combat this condition the New York State Highway Department began about 4 yr. ago to experiment with a blend of portland and natural cement for concrete pavement construction.

Preliminary tests made in the laboratory of the New York Department of Public Works indicated that the resistance of concrete to alternate freezing and thawing in a solution of calcium chloride was materially increased when a portion of the portland cement was replaced by natural cement. These preliminary laboratory experiments were followed by the construction of a number of experimental sections on actual pavements, using portland cements of various chemical compositions and two natural cements, both made in New York State and both readily available commercially.

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The Bureau of Public Roads became interested in this work not only from the abstract technical viewpoint but also because of a request from the Division of Highways of the New York Department of Public Works for permission to conduct certain of its field experiments on pavements financed either wholly or in part with Federal funds. It was felt that before approving its use on any extended mileage of highway very definite information should be obtained regarding the effect of the blend on other essential qualities, such as strength, volume change, etc., and, furthermore, that these tests should be made in the laboratory under closely controlled conditions.

The present paper reports the results of such a study as well as the results of a series of freezing-and-thawing tests on specimens taken from certain of the experimental pavements referred to above. The data on laboratory fabricated specimens are presented in two sections, the first dealing with tests of mortars and the second with concrete. The results of the freezing-and-thawing tests on concrete cores are discussed in a third section.

Tests on the mortar specimens included crushing strength, flexural strength, absorption, density, durability and volume change. Two portland cements, differing considerably in chemical composition, and two sands of widely different mineral composition were used in this portion of the investigation. Tests on concrete specimens fabricated in the laboratory included crushing strength, flexural strength and modulus of elasticity, using the same two portland cements with local fine and coarse aggregate. One brand only of natural cement was used in all specimens fabricated in the laboratory.

The freezing-and-thawing tests discussed in the third part of the report were made on cores take from four of the experimental concrete pavements referred to above. Two natural cements as well as portland cements conforming to four different classifications as to compound composition were used in these roads. Core specimens were taken from sections in which straight portland cement was used as well as from sections containing blends in the proportion of 6 parts portland to 1 part natural and 5 parts portland to 2 parts natural, by weight. These specimens were subjected to alternate freezing and thawing in water and in a 10 per cent solution of commerical calcium chloride.

MATERIALS USED IN LABORATORY FABRICATED SPECIMENS

The two portland cements included one brand from the Hudson River Valley region, designated as cement A, and one from the Lehigh Valley of Pennsylvania, designated as cement B. Both cements satisfactorily met the requirements for portland cement of the Society. The natural cement represented material produced in the northeastern part of the United States and is typical of the old type produced in vertical kilns. The fineness as determined by the Wagner turbidimeter was about the same for both portland cements, but the natural cement showed a fineness more nearly corresponding to that of high-early-strength cement. The results of the physical and chemical tests on the three cements are shown in Table I.

Three concrete sands were used in the investigation, sands A and C being composed essentially of quartz, and sand B consisting essentially of shale and quartz particles with appreciable amounts of limestone, slate, and sandstone. The sieve analyses and physical properties of the three sands and the coarse aggregate used in the tests are given in Table II. The coarse aggregate used in the laboratory fabricated concrete specimens was a limestone of known satisfactory quality and graded uniformly from No. 4 to $1\frac{1}{2}$-in. sieves.

TABLE I.—PROPERTIES OF CEMENTS USED IN LABORATORY FABRICATED SPECIMENS.

	Cement A	Cement B	Natural Cement
PHYSICAL PROPERTIES			
Apparent specific gravity	3.14	3.11	3.00
Specific surface, sq. cm. per g.	1730	1805	2680
Retained on No. 200 sieve, per cent	7.1	9.6	10.5
Normal consistency, per cent	23.7	24.0	26.0
Time of set: Initial	3 hr. 15 min.	2 hr. 55 min.	1 hr. 5 min.
Final	5 hr. 5 min.	4 hr. 55 min.	1 hr. 45 min.
Tensile strength, lb. per sq. in.			
7 days	380	370	50
28 days	445	445	150
CHEMICAL ANALYSES			
Silica, per cent	22.41	20.80	24.91
Alumina, per cent	4.78	6.47	9.65
Iron, per cent	3.72	2.38	
Lime, per cent	64.55	63.22	33.93
Magnesia, per cent	1.15	3.45	19.92
Sulfuric anhydride, per cent	1.72	1.77	1.42
Loss on ignition, per cent	1.37	1.47	8.55
COMPUTED COMPOUND COMPOSITIONS			
Tricalcium silicate, per cent	50	47	....
Dicalcium silicate, per cent	26	25	....
Tricalcium aluminate, per cent	6	13	....
Tetracalcium alumino-ferrite, per cent	11	7	....

OUTLINE OF MORTAR TESTS

For the various tests on mortars, each cement was used with sands A and B, making four combinations of materials. Specimens were made using 100 per cent portland cement, and blended cements in which 14 and 28 per cent by weight of portland cement was replaced by natural cement. These values corresponded to the replacements used on the New York experimental roads, which were of the order of 1 sack or 2 sacks in a 7-sack batch, or 14 and 28 per cent, respectively.

TABLE II.—PROPERTIES OF AGGREGATES.

SIEVE ANALYSES OF FINE AGGREGATES

	Sand A	Sand B	Sand C
Retained on $\frac{3}{8}$-in. sieve, per cent	0	0	0
Retained on No. 4 sieve, per cent	2	4	7
Retained on No. 8 sieve, per cent	11	21	24
Retained on No. 16 sieve, per cent	27	45	39
Retained on No. 30 sieve, per cent	49	67	53
Retained on No. 50 sieve, per cent	84	87	86
Retained on No. 100 sieve, per cent	97	95	97
Fineness modulus	2.70	3.19	3.06

PHYSICAL PROPERTIES

	Sand A	Sand B	Sand C	Coarse Aggregate
Absorption, per cent	0.3	2.2	0.9	0.32
Organic matter	OK	OK	OK	....
Decantation loss, per cent	1.4	2.5	1.5	....
Compressive strength ratio:				
7 days	140	123	121	....
28 days	130	124	121	....
Wear, per cent, Deval	....	....	....	3.0

For each cement, a sufficient quantity of water was added to the straight portland cement – sand A mixture to give a flow of approximately 90 on the 10-in. flow table. The same quantity of water was then used in all other mixtures containing this cement. This resulted in slightly lower flows (drier mortars) for the mixtures containing sand B and also somewhat lower flows for the mixtures containing natural cement. All consistencies, however, were plastic and workable, in so far as could be determined when fabricating the test specimens. The consistencies of the various mixes, as determined by the use of the 10-in. flow table, are shown in Table III.

An outline of the various tests made in the mortar series is given below:

1. Crushing and flexural tests on 2-in. cubes and 2 by 3 by 18-in. beams, using a mix of 1:2 by weight. Specimens were cured continuously in water at 70 F., and broken at ages of 7, 28, 90, 180 and 360 days. Duplicate sets of specimens were cured in air (with 7 days initial water cure) and broken at 28, 90, 180 and 360 days. Beam specimens were broken as cantilevers, using portions of the same beam for tests at various ages.

TABLE III.—EFFECT OF BLENDING PORTLAND AND NATURAL CEMENT UPON THE CONSISTENCY OF MORTARS AS DETERMINED BY THE 10-IN. FLOW TABLE.

Portland Cement Replaced by Natural Cement, per cent	Beams	Cubes	Beams	Cubes
	Cement A - Sand A		Cement B - Sand A	
0	91	93	93	93
14	87	90	90	89
28	83	83	88	85
	Cement A - Sand B		Cement B - Sand B	
0	83	83	85	85
14	79	76	82	80
28	74	70	79	78

2. Tensile strength briquets, using standard Ottawa sand with a 1:3 mix by weight. Specimens were broken at 7 and 28 days.

3. Tests for volume change on 2 by 3 by 18-in. beams, using 1:2 mix by weight. One set of specimens was stored in air for 120 days, the other continuously in water at 70 F. for 180 days. As a supplementary portion of this phase of the work, after 180 days water storage, the beams were frozen and thawed for 6 cycles, dried at 120 F. for 4 days, and resaturated in water at 70 F. for 3 days when the freezing-and-thawing cycle was again started. In this test, measurements of length were made at the conclusion of each 3-day resaturation period. A view of the linear comparator used in all measurements of volume change is shown in Fig. 1.

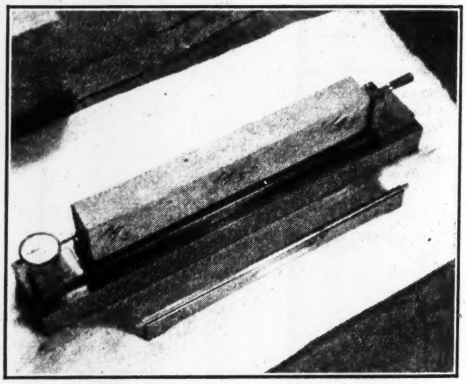


FIG. 1.—View of Horizontal Comparator Used in Measuring Length Change of Mortar Beams.

4. Miscellaneous tests included absorption and bulk specific gravity tests on 2 by 4-in. cylinders of 1:2 mortar, sodium sulfate soundness tests on 1 by 1 by 6-in. bars, using a mix of 1:5 by weight of Ottawa sand, and autoclave tests on 1 by 1 by 10-in. neat cement bars.

DISCUSSION OF TEST RESULTS

Strength Tests:

The results obtained in the strength tests of mortars are given in Figs. 2 to 5. The values have been plotted so as to show on the left the average results for the two sands with each cement and on the right the average results for the two cements with each sand.

Fig. 2 gives the results of crushing tests on specimens continuously water cured. When the results are averaged it will be seen that, for all combinations of materials, the straight portland cement mixes developed the greatest strength at all ages. With the exception of the 180- and 360-day values for the straight portland cement mortar in which sand A was used with cements A and B, all mortars exhibited gain in strength from the 7-day up to the 360-day periods.

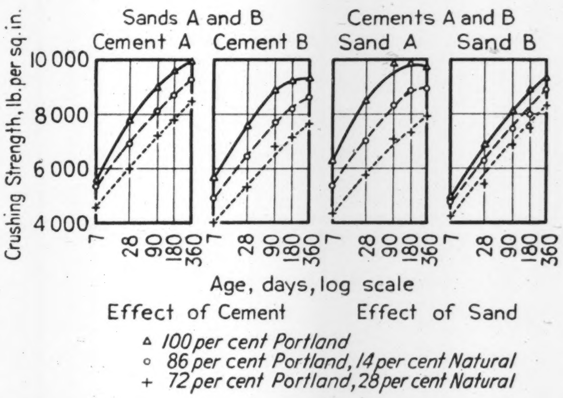


Fig. 2.—Effect of Blending Portland and Natural Cement on the Crushing Strength of 2-in. Mortar Cubes Cured in Water.

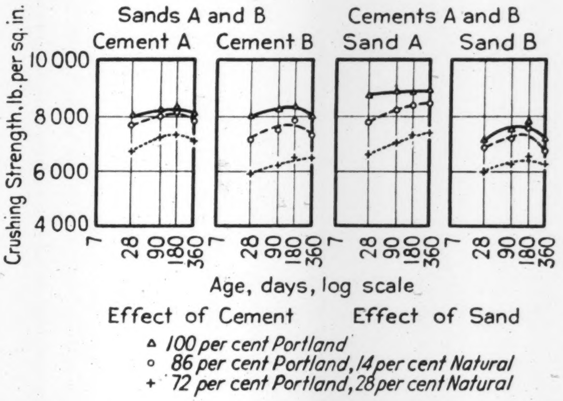


Fig. 3.—Effect of Blending Portland and Natural Cement on the Crushing Strength of 2-in. Mortar Cubes Cured in Air.

The effect of cement is indicated in the left-hand portion of Fig. 2. Here it may be seen that cement A gave slightly greater strengths than cement B. In the right-hand portion of Fig. 2 is shown the effect of changing the sand. In the unblended mix, as well as in the mortar containing the 14 per cent replacement, sand A showed greater strength at all ages. However, in the mix containing the 28 per cent replacement, the strengths were about the same. In other words, replacing portland cement with natural cement seemed to affect the strength of mortars made with sand A much more than when sand B was used.

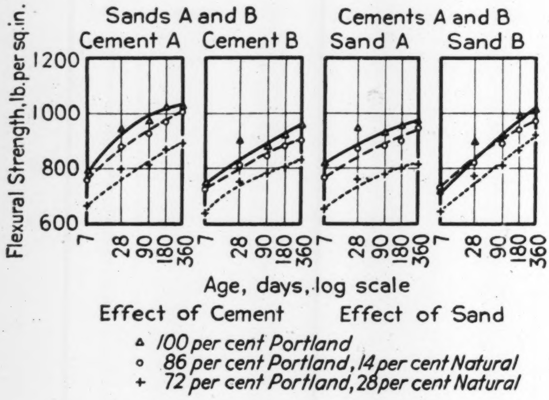


FIG. 4.—Effect of Blending Portland and Natural Cement on Flexural Strength of 18-in. Mortar Beams Cured in Water.

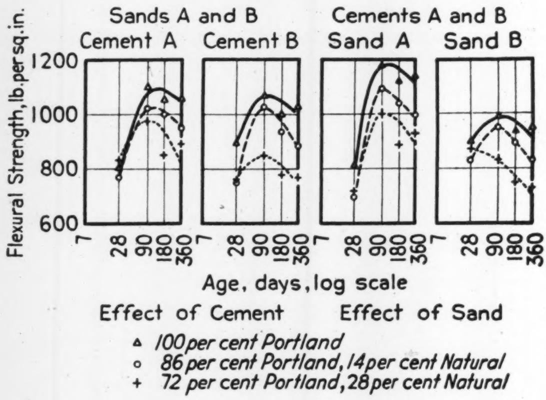


FIG. 5.—Effect of Blending Portland and Natural Cement on Flexural Strength of 18-in. Mortar Beams Cured in Air.

The values obtained on air-cured 2-in. cubes (after 7 days initial water storage) are given in Fig. 3. The average values show, in all cases, higher strengths for the mixes containing straight portland cement. Comparing Figs. 2 and 3 it will be noted that the specimens moist cured 7 days and air dried for 21 days were in general higher in strength than those cured continuously in water for 28 days. Further air storage, however, resulted in only slight gain in strength at 90 days, with little or no gain after that period.

Reference to Fig. 3 will show the effect of the cement and sand upon crushing strength of the air-dried specimens. Cement A exhibited slightly greater strength in the blends, with the unblended mortars having about the same strength at all ages. Sand A shows definitely higher strengths throughout. The most interesting point in connection with this figure is the decrease in strength caused by the replacement of portland cement with 28 per cent natural cement. The strengths obtained with this combination ran about 1000 to 2000 lb. per sq. in. lower than the straight portland cement mixes for the different cements and sands.

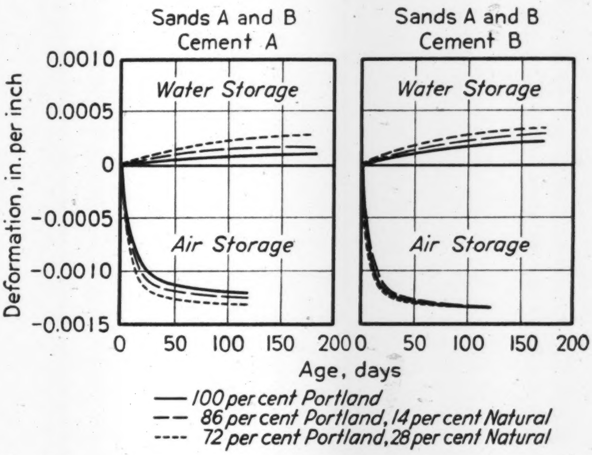


Fig. 6.—Effect of Blending Portland and Natural Cement on Volume Change of 18-in. Mortar Beams (Measured as Change in Linear Dimensions).

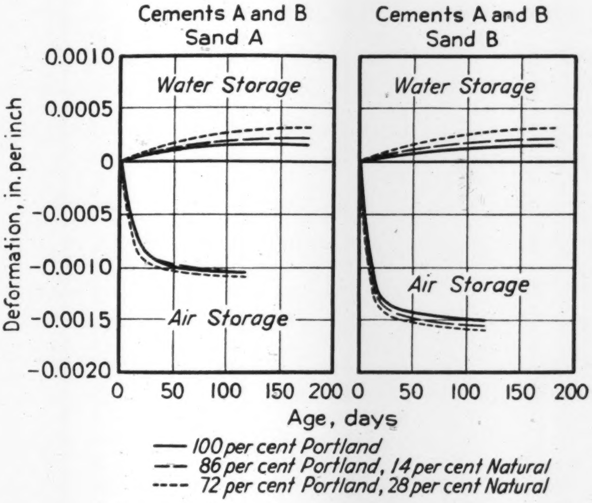


Fig. 7.—Effect of Blending Portland and Natural Cement on Volume Change of 18-in. Mortar Beams (Measured as Change in Linear Dimensions).

The effect of blending portland and natural cement upon the flexural strength of water-cured mortar beams is shown in Fig. 4. It will be noted that when the results are averaged as in Fig. 4 the straight portland cement mixes, with a single exception, produced the greatest strength at all ages. As in the water-cured compression specimens, the gain in strength was fairly uniform. However, the reduction in strength due to blending was not proportional to the amount of natural cement used, the 28 per cent replacement causing a much greater proportional reduction than the 14 per cent replacement. Cement A in general produced greater strengths for all combinations of materials than did cement B.

The results of flexure tests on air-cured beams are given in Fig. 5. Again it will be observed that here also with a single exception the straight portland cement mixes produced the greatest strength at all ages. With few exceptions the highest strengths in all cases were obtained at 90 days, all mixes showing retrogression after that period. In every case, the strengths at 90 days were higher than those of the corresponding water-cured specimens.

Volume Change of Mortar Beams:

The effect of blending portland and natural cement upon volume change (measured as change in linear dimension) of 18-in. mortar beams in continuous water storage is shown graphically in the upper portion of Figs. 6 and 7. The expansion is shown in 0.0001 in. per inch at the age indicated on the abscissas. It will be noted that a replacement of 28 per cent natural cement resulted in the greatest expansion at 180 days, and that the straight portland cement mixes for all combinations exhibited the least expansion. It is of interest to note the effect caused by the two different cements (Fig. 6). Cement B showed slightly greater expansions for both blended and unblended mixtures than cement A. However, as will be seen from Fig. 7, changing the sand had very little effect.

Volume changes resulting from continuous air storage is shown in the lower portions of Figs. 6 and 7. The treatment of these beams consisted of storage for 24 hr. in the moist closet, at the end of which time an initial reading was taken, followed by continued storage in laboratory air. From 90 to 120 days the beams were placed in a cabinet in which the air was dried with calcium chloride to decrease further the amount of moisture in the specimens. As expected, the beams in all cases showed slightly increased shrinkage.

Sands A and B when used with cement A gave approximately the same amount of shrinkage as when used with cement B (Fig. 6). However, when cements A and B were used with sand B, the total shrinkage at 120 days was about 50 per cent greater than when sand A was used (Fig. 7). Reference to the figures will show that although the initial shrinkage was somewhat greater in the case of the blended cements, the total shrinkage at the end of 120 days continuous air storage was about the same.

Accelerated Weathering Tests:

The mortar beams from the continuous water storage series were measured for length at the end of 180 days, and were then subjected to 15 rounds of the following accelerated weathering treatment. Each round required 13 days for completion and consisted of 6 cycles of freezing and thawing, each 24 hr. in duration, followed in turn by drying for 4 days in an electric oven at 120 F. and resaturation for 3 days in storage water at 70 F. Readings were taken on each beam at the end of the resaturation period and were discontinued at the end of the fifteenth round, at which time surface disintegration of the specimens had progressed to a point which affected the accuracy of the readings due to loosening of the gage points. Unit length changes at the end of the first, third, sixth, ninth, twelfth and fifteenth rounds are shown in Figs. 8 and 9. Here the data have been averaged in the same manner as in the previous figures so as to bring out the effect of variations in the cement and sand.

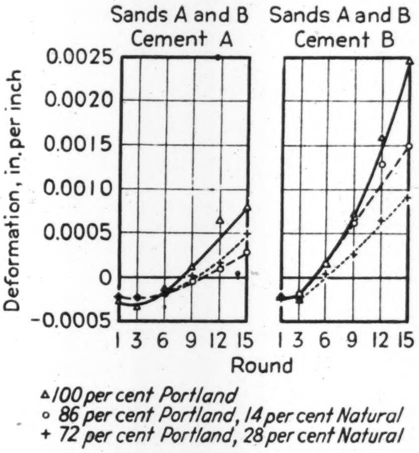


FIG. 8.—Effect of Freezing and Thawing, Followed by Drying, Cooling and Resaturation on Volume Change (Measured as Change in Linear Dimensions).

There are several points of interest in connection with these figures. In the first place, all combinations show an initial contraction followed by expansion up to the fifteenth round. The amount of initial contraction seemed to have been affected greatly by the combination used, being greatest for the combinations involving cement A with the two sands (Fig. 8) and sand B with the two cements (Fig. 9). The combinations involving sand A with cements A and B showed very little initial contraction. This effect may be due to the drying cycle which was introduced. Drying at 120 F. for 4 days undoubtedly induced high shrinkage in the cement paste, subjecting the particles of aggregate to high compressive stress. It is possible that the difference in the ability of the sands to resist this stress without permanent deformation may have accounted for the residual shrinkage observed in the case of the specimens containing sand B which was composed of comparatively low-strength material. The quartz particles composing sand A may have possessed sufficient strength to resist these shrinkage stresses without permanent deformation, so that the specimens when resaturated returned practically to their original length. The accumulated effect of this action may account also for the relatively small expansions observed with

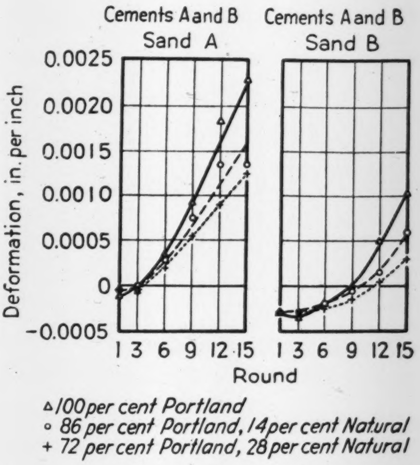


FIG. 9.—Effect of Freezing and Thawing, Followed by Drying, Cooling and Resaturation on Volume Change (Measured as Change in Linear Dimensions).

sand B as compared to the expansions shown with sand A.

It will be observed also that, in all cases, specimens containing the blended cement showed lower expansions than the corresponding specimens containing 100 per cent portland cement. It is usually assumed, in so far as expansion is concerned, that repeated freezing and thawing produces a gradually expanding paste, resulting in the eventual disintegration of the specimen. On this basis, the use of the blend may be considered beneficial. However, visual examination of the specimens as the test proceeded failed to reveal any consistent difference in appearance at any stage between the specimens containing the blend and those containing the straight portland cement. So far as could be determined visually by inspection of the surface, all of the combinations failed at about the same rate. All were badly exfoliated or surface pitted by the end of the fifteenth round and apparently possessed little strength.

TABLE IV.—EFFECT OF BLENDING PORTLAND AND NATURAL CEMENT ON TENSILE STRENGTH.

Mix 1:3—Standard Ottawa sand.

NOTE.—Figures in parentheses indicate percentage of the strength of 100 per cent portland cement mixes.

Each value is the average of 15 tests.

Portland Cement Replaced by Natural Cement, per cent	Tensile Strength, lb. per sq. in.			
	Cement A		Cement B	
	7 days	28 days	7 days	28 days
0	390	455	390	450
14	370 (95)	450 (99)	360 (92)	440 (98)
28	345 (88)	405 (89)	330 (85)	405 (90)

Miscellaneous Tests of Mortars:

The effect of replacements with natural cement on the tensile strength of 1:3 Ottawa sand briquets is shown in Table IV. It is of interest to note that, for both cements, the 14 per cent replacement at 28 days had very nearly the same strength as the straight portland-cement mortar. For 28 per cent replacement the strengths at 28 days were approximately 90 per cent of the straight portland.

The effect of the blends upon the bulk specific gravity and absorption of mortar specimens is shown in Table V. Cylinders 2 by 4 in. in size of 1:2 mortar, after 24 hr. in moist air, were stored in water at 70 F. and the bulk specific gravity and absorption determined after 7 and 28 days by first determining the saturated-surface dry weights of the specimens in air and in water and then drying to constant weight. It will be observed that blending with natural cement lowered the bulk specific gravity and increased the absorption in all cases.

TABLE V.—EFFECT OF BLENDING PORTLAND AND NATURAL CEMENT ON BULK SPECIFIC GRAVITY AND ABSORPTION OF 2 BY 4-IN. MORTAR CYLINDERS.

Mix 1:2 by weight—water cure.

NOTE.—Figures in parentheses indicate percentage of the corresponding values for 100 per cent portland cement mixes.

Each value is the average of 3 tests.

Portland Cement Replaced by Natural Cement, per cent	Bulk Specific Gravity		Absorption, per cent	
	7 days	28 days	7 days	28 days
CEMENT A - SAND A				
0	2.10	2.12	9.52	9.23
14	2.07 (99)	2.09 (99)	9.89 (104)	10.01 (108)
28	2.03 (97)	2.04 (96)	10.32 (108)	10.59 (115)
CEMENT A - SAND B				
0	2.09	2.10	10.49	10.32
14	2.07 (99)	2.08 (99)	10.84 (103)	10.73 (104)
28	2.04 (98)	2.05 (98)	11.34 (108)	11.19 (108)
CEMENT B - SAND A				
0	2.07	2.11	10.03	9.98
14	2.05 (99)	2.06 (98)	10.66 (106)	10.62 (106)
28	2.02 (98)	2.02 (96)	11.27 (112)	11.40 (114)
CEMENT B - SAND B				
0	2.09	2.07	10.95	10.76
14	2.04 (98)	2.06 (100)	11.34 (104)	11.11 (103)
28	2.01 (96)	2.04 (99)	11.73 (107)	11.57 (108)

The decrease in density indicated by these tests may have been due to a slight increase in the air voids brought about by the use of natural cement. Other tests have indicated that air voids in concrete are increased somewhat by using the blend.

Bars, 1 by 1 by 6 in. in section, using a 1:5 standard Ottawa sand mortar, and 0, 14 and 28-per-cent replacement with natural cement, were fabricated and stored for 6 days in water at 70 F. At the end of this period the bars were measured for length and then immersed in a 10 per cent solution of sodium sulfate. After 21 days of continuous storage in this solution at 70 F., they were again measured for length. The amount of expansion was read to 0.001 in. The results are shown in Fig. 10 (left panel). Each value is the average of three tests. It will be noted that cement B showed greater expansion in all cases than cement A and also that the replacements with natural cement reduced the amount of expansion in both cases. These tests seem to indicate that blending with natural cement increases resistance to sulfate action.

The expansions of neat cement bars exposed for 3 hr. at 420 F. in the autoclave are shown in the right panel of Fig. 10. In both cases, the replacement of 14 and 28 per cent portland by natural cement resulted in increased expansion. It will be noted, however, that cement B, unblended, showed considerably greater expansion than cement A with a 28 per cent replacement of natural cement. The relatively high expansions shown by cement B may possibly be associated with the higher magnesia and greater C_3A content of this cement (see Table I).

Tests of Concrete Specimens Fabricated in the Laboratory:

The same two portland cements and the same natural cement used in the mortar tests were combined with a local fine and coarse aggregate in fabricating the concrete specimens. The characteristics of the aggregates are shown in Table II. Sand C was used in the concrete tests. The coarse aggregate was uniformly graded from No. 4 to 1½-in. sieves. Two replacements were used, 14 per cent and 28 per cent as in the mortar tests.

A typical concrete paving mix containing 6 sacks of cement per cubic yard with sufficient water for a 2½-in. slump was used. This resulted in a net water-cement ratio by volume of approximately 0.75.

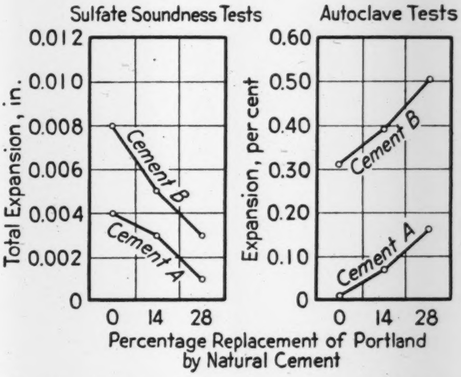


FIG. 10.—Effect of Blending Portland and Natural Cement upon Volume Change in Sulfate Solution and in the Autoclave (Measured as Change in Linear Dimensions).

Although the tests made on the mortars showed that the consistency of the mix was affected somewhat by the substitution of natural cement for portland cement, the slump of the concrete containing the blends was the same as that obtained with the straight portland cement. However, in manipulating the concrete it was found that the blended cement mixtures had a "stickiness" which was not apparent in the case of those containing the straight portland cement.

Flexure tests were made on beams 6 by 6 by 21 in. in size, loaded at the third points of an 18-in. span. Compression tests as well as the measurements for modulus of elasticity, using a Martens extensometer, were made on 6 by 12-in. cylinders. Five specimens were made for each test condition and tests were made at 7, 28, 90 and 180 and 360 days. All specimens were stored continuously in moist air at 70 F. until tested.

The results of the tests for strength and modulus of elasticity on laboratory fabricated concrete specimens are shown in Fig. 11.

Referring to the data for cement A (left panel of Fig. 11), it will be observed that there was a gradual increase in crushing strength with age for the unblended as well as for the blended cement. Blending with natural cement decreased the average strength at all ages about 1 per cent in the case of the 14 per cent replacement and about 8 per cent in the case of the 28 per cent replacement. Similar data for cement B are shown in the next panel to the right. In the case of the straight portland cement it will be observed that the same order of strengths was obtained as in the case of cement A. However, with the blends the reduction in strength as compared to the straight portland was more marked, the corresponding figures being 7 per cent for the 14 per cent replacement and 17 per cent for the 28 per cent replacement.

It is interesting to observe that the 14 per cent blends for both cements A and B exhibited higher strengths at 360 days than the corresponding straight portlands. This was due to the fact that the straight portland cement concretes showed no increase in strength beyond the 180-day period in the case of cement A, while for cement B no increase was observed after the 90-day period. On the other hand, the concrete containing the blended cements showed a fairly uniform increase in strength up to 360 days.

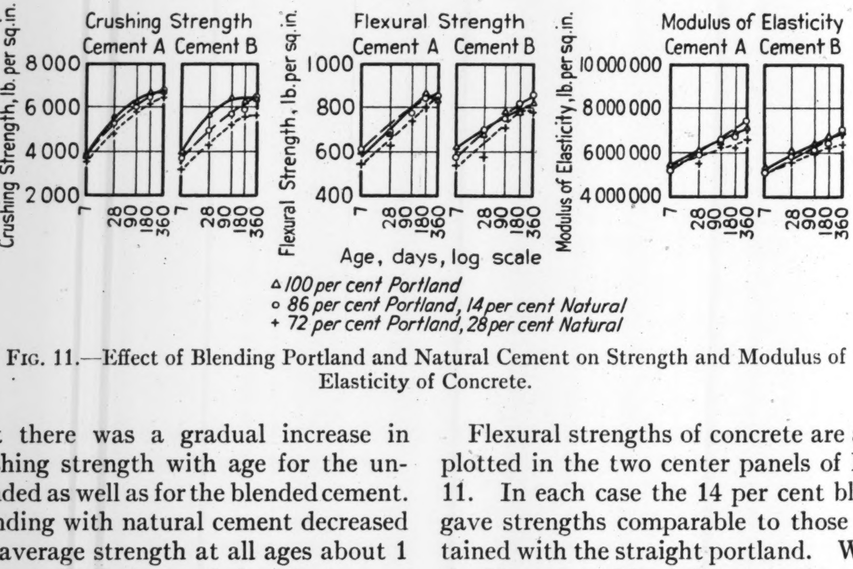


FIG. 11.—Effect of Blending Portland and Natural Cement on Strength and Modulus of Elasticity of Concrete.

Flexural strengths of concrete are also plotted in the two center panels of Fig. 11. In each case the 14 per cent blend gave strengths comparable to those obtained with the straight portland. With the 28 per cent blend the strengths averaged about 95 per cent of the straight portland. It is to be observed also that in the case of both portland cements the blended cement concrete showed higher strength ratios in flexure than in compression.

Results of tests for modulus of elasticity are shown in the two right panels of Fig. 11. Here again the addition of the natural cement is reflected in the somewhat lower results obtained with the blends.

In general, it may be said that the values obtained in flexure and modulus of elasticity for the 28 per cent replacement were about 95 per cent of those obtained for the straight portlands. The strength ratios in compression were not the same for both cements, cement B giving an average ratio of 83 per cent, which was about 10 per cent lower than that obtained for cement A. Comparing the concrete tests with the corresponding mortar tests it will be observed that replacement with the natural cement had a much greater effect on mortar strength than on concrete strength. This would, of course, be expected, due to the higher cement content in the mortar specimens.

Freezing-and-Thawing Tests of Cores Taken from Pavements:

The results of freezing-and-thawing tests on cores taken from four experimental projects in New York State are included below. On each of these projects specimens of straight portland-cement concrete and blended-cement concrete were taken from sections containing cement conforming to each of the four following classifications as to compound composition:

Specification Designation	Tricalcium Aluminate, C_3A, per cent	Tricalcium Silicate, C_3S, per cent
935	Not over 9	Not over 35
950	Not over 9	Between 48 and 52
1439	Not less than 14	Not over 39
1450	Not less than 14	Between 48 and 52

Natural cement of the same brand as used in the laboratory tests was used on two of the projects. The other two contained natural cement from another mill in New York State. A 6:1 blend was used on two of the jobs, a 5:2 blend on the other two.

This portion of the work was done in order to test specimens of concrete which had been subjected to the usual field manipulation during placing in order to study the effects of the "bleeding" or "water-gain" which frequently occurs during the finishing operation. This action results in the accumulation of an excess amount of water in the top surface of the pavement, leaving a weak, porous layer which is lacking in resistance to the disintegrating effects of ice and chemical salts.

In a preliminary series of freezing-and-thawing tests made on full depth cores it was found that disintegration was confined almost entirely to the top 2 in. of the specimen. Because of this, and also because of the saving in space which would result, it was decided to study the effect of the blend by testing the upper portion of the concrete only. Disks 2 in. in thickness were accordingly sawed from the tops of the cores. One set of these disks was alternately frozen and thawed in a 10 per cent solution of calcium chloride, the other set in plain water. In addition, a few tests were made to determine the resistance of similar disks sawed from the lower surface of the cores.

It was found desirable in this work to use a minimum amount of the chloride solution and to freeze rapidly in order to keep the solution from separating into layers of different density. This was accomplished by using close-fitting containers and immersing these containers in the brine solution of the refrigerator. The minimum temperature obtained was approximately −10 F., one complete cycle of freezing and thawing being obtained each day. The time required to lower the temperature of the specimen to this minimum was approximately 10 hr. The calcium chloride solution was changed after 10 cycles of freezing and thawing. These time-temperature relations applied both to freezing in chloride and in water.

Periodic examinations were made of the specimens, these examinations consisting of weight loss determinations and visual inspections. It was found that the weight losses on the 2-in. disks were meaningless. For instance, a specimen which had not lost more than 2 per cent in weight would become unsound and disintegrate. While some scaling of the surface devloped, unsoundness in general was caused by failure of the bond between the cement and aggregate, resulting in the eventual disintegration of the entire specimen.

TABLE VI.—RESISTANCE OF CORE SPECIMENS TO FREEZING AND THAWING.

Rating of 100 indicates all 8 specimens sound.
Rating of 0 indicates all 8 specimens disintegrated.

Blend[a]	Rating at Number of Cycles Indicated					
	Calcium Chloride	Water				
	35 Cycles	35 Cycles	50 Cycles	75 Cycles	100 Cycles	150 Cycles
PROJECT A						
7-0	0	45	10	0	0	0
6-1G	90	95	90	90	90	90
PROJECT B						
7-0	25	50	30	25	25	25
5-2G	100	100	100	100	100	100
PROJECT C						
7-0	40	30	5	0[b]	0	0
6-1H	15	25	10	0[c]	0	0
PROJECT D						
7-0	0	10	5	0	0	0
5-2H	55	65	55	30	30	25

[a] Figures indicate the number of sacks of portland and natural cement in a 7-sack batch. Letters refer to the brand of natural cement.

[b] At 65 alternations.

[c] At 60 alternations.

For each project and for each type of freezing there were two groups of 8 specimens each. In one group there were 4 pairs of specimens containing straight portland cement and 4 pairs containing the same portland cements blended with natural cement in the proportion of either 6:1 or 5:2. The 4 pairs of specimens in each group represented concrete containing the 4 classes of portland cement mentioned above.

In order to evaluate the resistance to freezing and thawing, the individual specimens were examined at various intervals and rated in accordance with their appearance and ring when struck with a hammer. A specimen which was entirely sound was given a rating of 12½. An unsound specimen was rated at 2½, while a specimen which appeared to be between these two conditions was considered as questionable and was given a rating of 7½. Any specimen which had disintegrated was given a rating of zero. If all of the specimens of a particular group were sound the rating would be 100. On the other hand, if all of the specimens had disintegrated, the rating would be zero. By this method of rating, the results secured with the four cements used on each project were averaged for comparison with the results obtained with the same portland cements blended with natural cement. This procedure was decided upon after a study of the individual results had failed to reveal any consistent relation between the composition of the portland cement and its resistance to freezing and thawing either when used straight or when blended. With a few exceptions, concrete made from portland cements conforming to the various composition classifications, when used on a given job, behaved about alike in so far as resistance to freezing and thawing was concerned.

The ratings of the several groups of specimens after 35 alternations in a 10 per cent solution of calcium chloride, as well as the corresponding ratings when frozen in water for various periods up to 150 alternations, are given in Table VI.

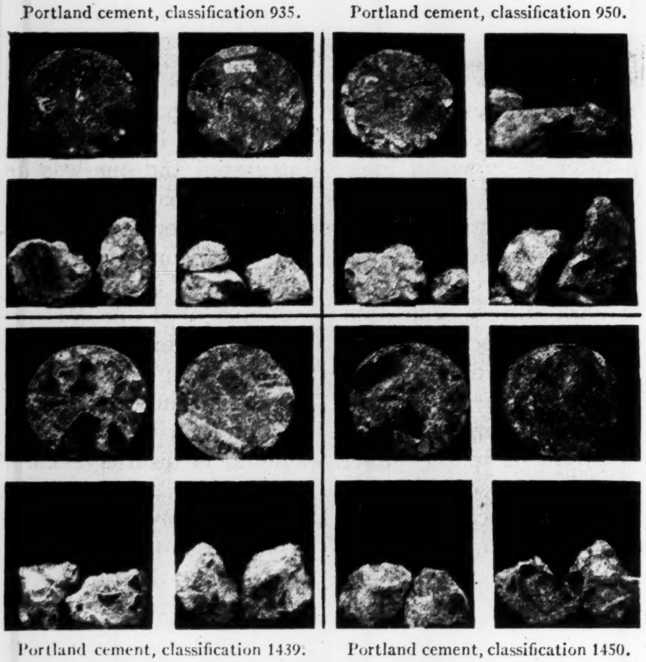
The appearance of each individual specimen at the end of 35 alternations in calcium chloride and at the end of 50 alternations in water is shown in Figs. 12-19 inclusive. In each figure the specimens are arranged as follows: The four specimens shown in the upper left quadrant contain portland cement conforming to New York State specification classification 935, the two specimens in the top row containing the blended cement and the two specimens in the second row the straight portland cement. Similarly, the four specimens in the upper right quadrant contain cement conforming to the 950 classification, those in the lower left quadrant the 1439 cement and those in the lower right quadrant the 1450 cement. It will be noted that all specimens shown in the first and third rows in each photograph are blended cement concrete and those in the second and fourth rows are straight portland-cement concrete.

Referring to Table VI and to Figs. 12–15, inclusive, it will be noted that the use of the blend resulted in a marked increase in resistance to freezing in calcium chloride in the case of projects A and B, some improvement in the case of project D and no improvement in the case of project C. Projects A and B contained natural cement G in the blend, whereas natural cement H was used in the case of projects C and D.

A study of the individual results show that in the case of project A all eight of the straight portland-cement specimens had disintegrated, while seven of the blended cement specimens were still sound. In the case of project B, six of the eight straight portland-cement specimens had disintegrated, whereas all eight of the blended specimens were sound. In project C five of the unblended specimens had disintegrated as compared to six of the specimens containing the blended cement. In the case of project D all eight of the unblended specimens had disintegrated, whereas three of the blended specimens had disintegrated and one was unsound.

The comparative effect of freezing in calcium chloride and in water may be noted by comparing the ratings at 35 alternations as shown in the first two columns of Table VI. It will be observed that in the case of projects A and B the action in plain water was less severe than in calcium chloride. However, in the case of projects C and D the amount of disintegration was about the same for both types of freezing. The comparatively low resistance of projects C and D to freezing in water would indicate that the quality of the concrete was inferior to that in projects A and B.

The study of the comparative behavior of the various specimens when frozen in water for periods beyond 35 cycles may also be made by referring to Table VI. In the case of projects A and B, virtually the same resistance is shown for the the blended cements when frozen in water up to 150 cycles as was indicated when the specimens were frozen in calcium chloride for 35 cycles. However, it will be observed that the straight portland-cement concrete in project B was considerably more resistant to freezing and thawing in water at periods beyond 35 cycles than was the concrete in project A. The concrete in project C appeared to be definitely inferior, all specimens in the case of both the straight portland and the blend having disintegrated at either 60 or 65 cycles. In the case of project D the behavior of the straight portland-cement concrete specimens also indicated comparatively inferior concrete as compared to projects A and B. However, the blended specimens from project D were considerably more resistant than those containing straight portland cement although, as

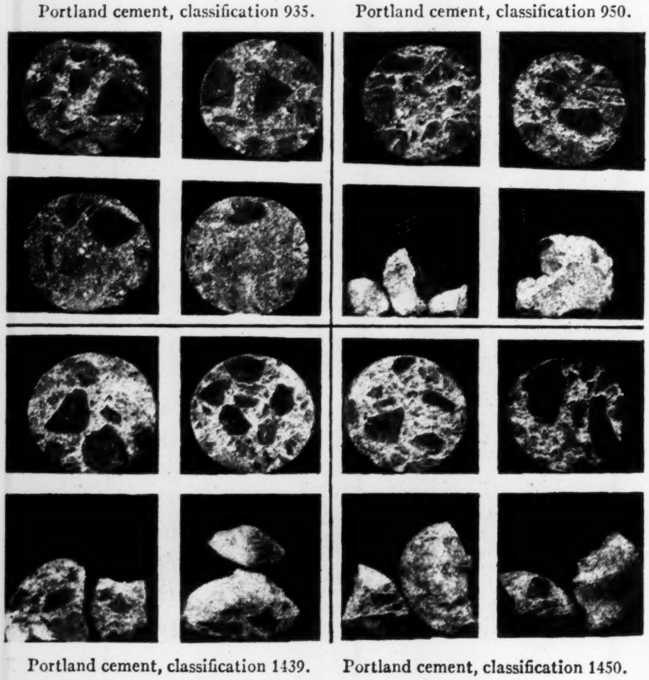


Portland cement, classification 935. Portland cement, classification 950.

Portland cement, classification 1439. Portland cement, classification 1450.

FIG. 12.—Project A. Specimens After 35 Alternations of Freezing and Thawing in a 10-per-cent Solution of Calcium Chloride.

The top row of each quadrant is blended cement; the bottom row of each quadrant is straight portland cement.



Portland cement, classification 935. Portland cement, classification 950.

Portland cement, classification 1439. Portland cement, classification 1450.

FIG. 13.—Project B. Specimens After 35 Alternations of Freezing and Thawing in a 10-per-cent Solution of Calcium Chloride.

The top row of each quadrant is blended cement; the bottom row of each quadrant is straight portland cement.

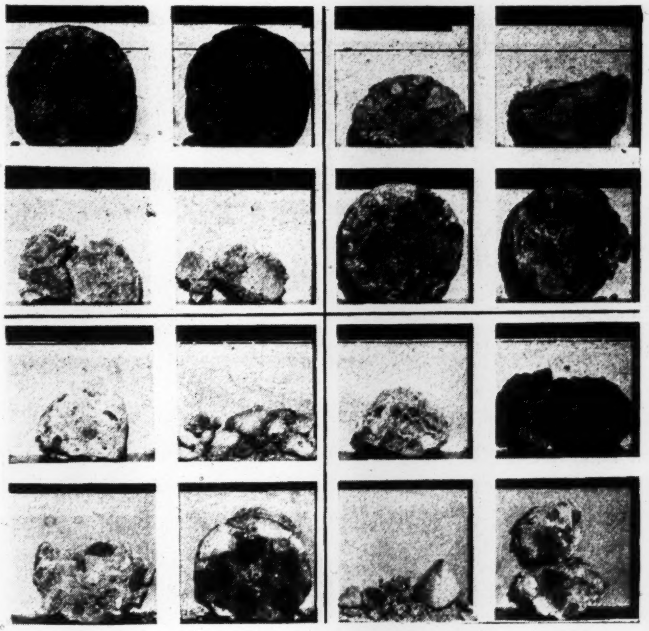

Portland cement, classification 935.
Portland cement, classification 950.
Portland cement, classification 1439.
Portland cement, classification 1450.

FIG. 14.—Project C. Specimens After 35 Alternations of Freezing and Thawing in a 10-per-cent Solution of Calcium Chloride.

The top row of each quadrant is blended cement; the bottom row of each quadrant is straight portland cement.

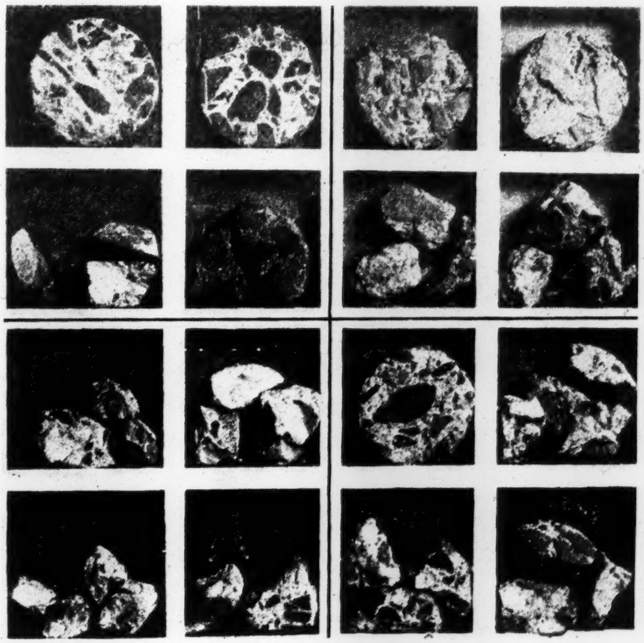
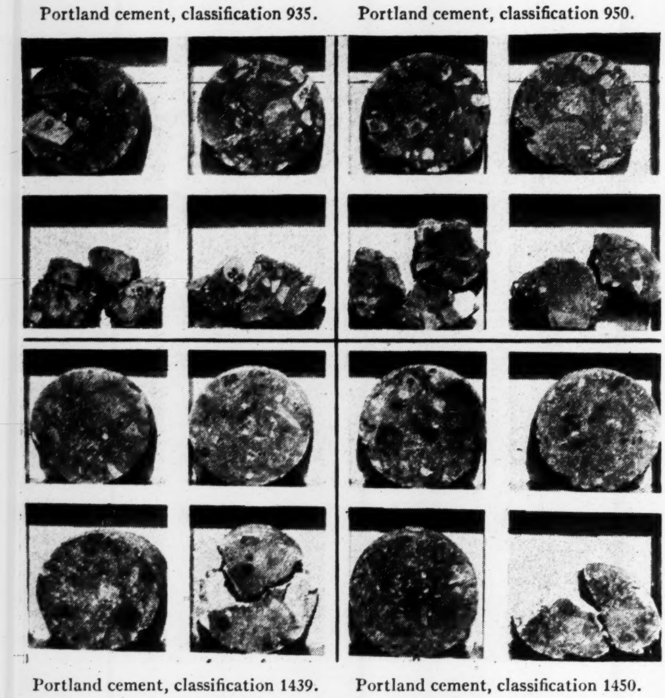

Portland cement, classification 935.
Portland cement, classification 950.
Portland cement, classification 1439.
Portland cement, classification 1450.

FIG. 15.—Project D. Specimens After 35 Alternations of Freezing and Thawing in a 10-per-cent Solution of Calcium Chloride.

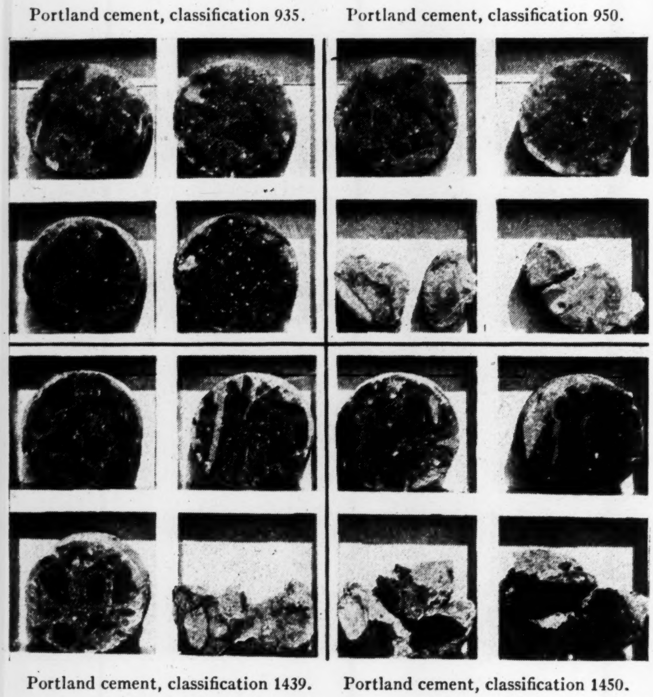
The top row of each quadrant is blended cement; the bottom row of each quadrant is straight portland cement.



Portland cement, classification 935. Portland cement, classification 950.

Portland cement, classification 1439. Portland cement, classification 1450.

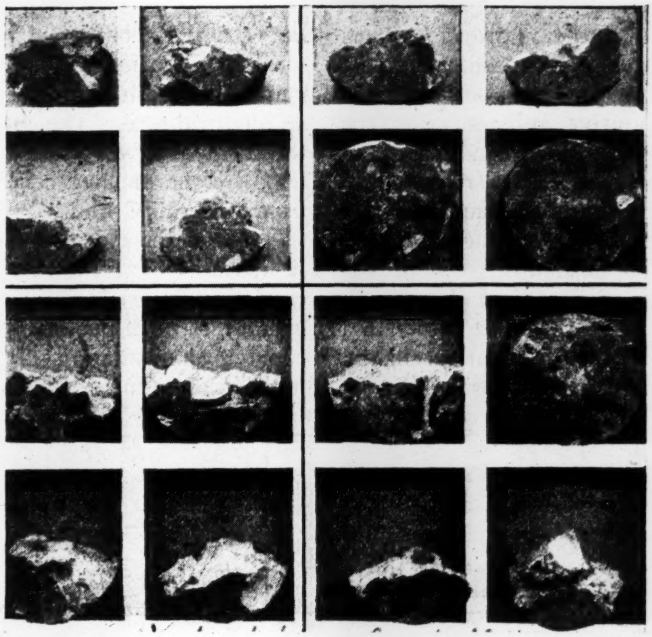
FIG. 16.—Project A. Specimens After 50 Alternations of Freezing and Thawing in Water.
The top row of each quadrant is blended cement; the bottom row of each quadrant is straight portland cement.



Portland cement, classification 935. Portland cement, classification 950.

Portland cement, classification 1439. Portland cement, classification 1450.

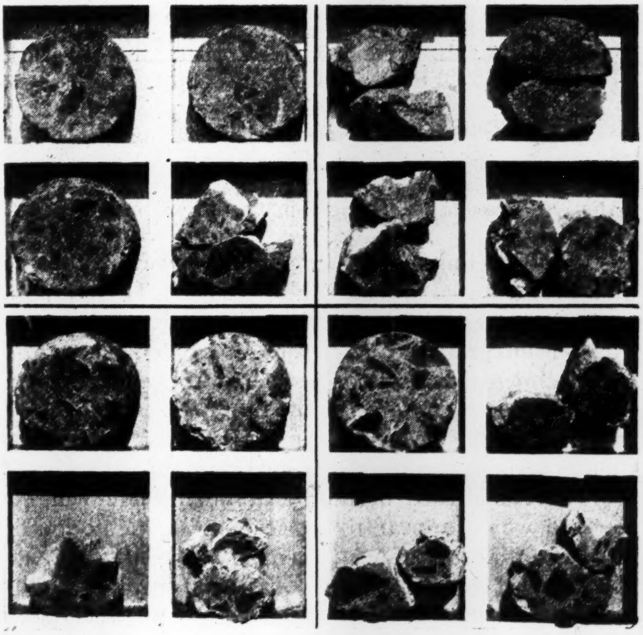
FIG. 17.—Project B. Specimens After 50 Alternations of Freezing and Thawing in Water.
The top row of each quadrant is blended cement; the bottom row of each quadrant is straight portland cement.



Portland cement, classification 935. Portland cement, classification 950.

Portland cement, classification 1439. Portland cement, classification 1450.

FIG. 18.—Project C. Specimens After 50 Alternations of Freezing and Thawing in Water.
The top row of each quadrant is blended cement; the bottom row of each quadrant is straight portland cement.



Portland cement, classification 935. Portland cement, classification 950.

Portland cement, classification 1439. Portland cement, classification 1450.

FIG. 19.—Project D. Specimens After 50 Alternations of Freezing and Thawing in Water.
The top row of each quadrant is blended cement; the bottom row of each quadrant is straight portland cement.

indicated above, the improvement was not as marked as in the case of projects A and B.

The comparative resistance of the individual specimens when frozen in water may be studied by referring to Fig. 20, which shows for each test disk the number of cycles at which the various stages of unsoundness were observed. Thus, for project A, cement classification 1439, the chart shows that of the two unblended cement specimens, one developed questionable soundness at 50 alternations, became definitely unsound at 60 and disintegrated at 75. The other specimen was questionable at 20 alternations and disintegrated at 25. The two blended specimens were sound at 150 alternations. It will be observed that there is no consistent relation between composition of cement and resistance to freezing. This has already been commented upon and would appear to justify the method of rating the groups as a whole as shown in Table VI.

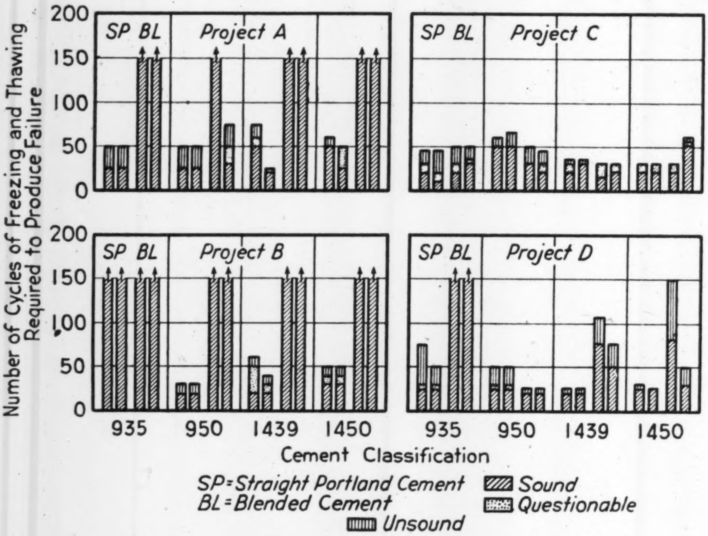


FIG. 20.—Results of Freezing Top 2 in. of Cores in Water.

An interesting feature of this chart is the close agreement observed in general between the behavior of the two specimens representing a given concrete. In many cases, failure progressed at almost an identical rate. The chart also shows the comparatively low resistance offered by the blended cement specimens from projects C and D. Whether this was due to the fact that natural cement H was used or to the fact that the concrete in these projects appeared, in general, to be of poorer quality than the concrete used in projects A and B cannot be determined definitely. The fact remains, however, that the use of natural cement H did not improve the concrete in project C at all and that while some improvement was shown for project D, the results were not so positive as those indicated for projects A and B, in both of which natural cement G was used.

Although there will be no extended discussion of the action of the blended-cement concrete as compared to concrete containing only portland cement, there were certain physical differences noted in the concrete at the time the specimens were fabricated which will be discussed briefly. It has been observed in the field that there is less bleeding or water gain in the case of the concrete made with the blended cement than there is when portland cement is used alone. This observation led to a short laboratory investigation, the purpose of which was to determine the amount of water accumulating on the top of the fabricated specimens.

The concrete was proportioned in accordance with the New York State Highway Specifications and contained approximately 6.3 sacks of cement per cubic yard. A slump of $2\frac{1}{2}$ inches was used, requiring a water-cement ratio of 0.70.

Test specimens consisted of 6 by 6 by 21-in. beams molded in the usual way. After molding, the tops of the specimens were exposed to the laboratory air with no curing. Two specimens were made using 100 per cent portland cement, two using a blend of 86 per cent portland and 14 per cent natural cement, and two using 72 per cent portland and 28 per cent natural cement.

At stated intervals after molding, the water which had accumulated at the top was carefully removed. The total water removed at the end of $2\frac{1}{2}$ hr. is shown in Table VII, expressed as a percentage of the free water going into the concrete. This table also gives the results of a second test run several days later. In this case the results show total water removed at the end of 4 hr.

From the results shown in the table it is evident that the substitution of natural cement for portland cement was effective in reducing bleeding. As will be noted, the bleeding was reduced as the amount of natural cement was increased, a replacement of 28 per cent resulting in a reduction of more than one-half. Assuming that bleeding leaves the upper layer of concrete porous and weak as compared to the lower structure, it is apparent that any material used or method of manipulation employed which will reduce bleeding should prove beneficial.

Data have been presented to show how the substitution of natural cement for portland cement materially increased the resistance to frost action of the top portions of the drilled cores.

In order to determine what effect water travel or bleeding has on durability, 2-in. disks were cut from the tops and also the bottoms of identical cores and frozen in a 10 per cent calcium chloride solution until failure occurred. Using nine disks in each case, it was found that the average number of alternations required to produce failure was 20 in the case of the tops of the cores and 35 in the case of the bottoms, a difference of 15 alternations. All of the specimens were taken from projects A and B and represented five different portland cements, none of which was blended with natural cement.

TABLE VII.—WATER REMOVED FROM TOPS OF 6 BY 6 BY 21-IN. BEAM SPECIMENS IN BLEEDING TESTS.

Cement Blend, per cent		Water Removed from Specimens, per cent	
Portland	Natural Cement G	First Test[a]	Second Test[a]
100	0	7.6	7.4
86	14	4.6	4.8
72	28	3.5	2.9

[a] Based on total free water in concrete.

SUMMARY OF RESULTS

In the summary given below reference to a 1- or 2-sack replacement of portland cement by natural cement corresponds to a 14 or 28 per cent replacement by weight, respectively. The average values for strength are the averages of results of tests at all ages.

(*a*) The results of tests on specimens

fabricated in the laboratory indicate that replacing a portion of the portland cement with natural cement:

1. Reduced the average crushing strength of 1:2 mortar specimens 7 per cent in the case of the 1-sack replacement and 19 per cent in the case of the 2-sack replacement (Figs. 2 and 3).

2. Reduced the average flexural strength of 1:2 mortar specimens 6 per cent in the case of the 1-sack replacement and 15 per cent in the case of the 2-sack replacement (Figs. 4 and 5).

3. Increased expansion of 1:2 mortar bars under continuous water storage up to 180 days (Figs. 6 and 7).

4. Slightly increased contraction of 1:2 mortar bars under air storage conditions up to 120 days (Figs. 6 and 7).

5. Considerably reduced the expansion of 1:2 mortar bars caused by repeated freezing and thawing, followed by heating, cooling and resaturation (Figs. 8 and 9).

6. Decreased the bulk specific gravity and increased the water absorption of 1:2 mortar specimens (Table V).

7. Reduced the expansion of 1:5 mortar bars when subjected to the action of a 10 per cent sulfate solution (Fig. 10).

8. Increased expansion of neat cement bars when subjected to the autoclave test at 420 F. for 3 hr. (Fig. 10).

9. Reduced the average crushing strength of concrete specimens containing 6 sacks of cement per cubic yard of concrete 4 per cent in the case of the 1-sack replacement and 12 per cent in the case of the 2-sack replacement (Fig. 11).

10. Increased the average flexural strength of concrete specimens containing 6 sacks of cement per cubic yard of concrete 1 per cent for the 1-sack replacement and reduced the flexural strength 6 per cent in the case of the 2-sack replacement (Fig. 11).

11. Decreased the average modulus of elasticity of concrete specimens containing 6 sacks of cement per cubic yard of concrete 1 per cent in the case of the 1-sack replacement and 5 per cent in the case of the 2-sack replacement (Fig. 11).

(*b*) The results of tests on cores taken from concrete pavements indicate that replacing a portion of the portland cement with natural cement considerably increased the resistance of the surface of the concrete to freezing and thawing in water and in a 10 per cent solution of calcium chloride (Table VI, Fig. 20).

In the tests covered by the above statement greater improvement in resistance was obtained by the use of natural cement G than by the use of natural cement H.

CONCLUSIONS

These tests indicate that, although the crushing and flexural strength of pavement concrete may be slightly reduced by the substitution for portland cement of 14 or 28 per cent natural cement G the resistance of the surface of the pavement to alternate freezing and thawing will be materially increased. No laboratory tests were made using natural cement H and tests of field specimens indicate that natural cement H did not improve the resistance of the concrete to the same extent as natural cement G.

DISCUSSION

Mr. Ira Paul[1] (*presented in written form*).—The differences noted in the laboratory chloride freezing-and-thawing tests between the natural cement G and natural cement H blends with portland cement were not detected in the specifications for natural cement. In our first specification the A.S.T.M. Standard Specifications for Natural Cement (C 10 - 09)[2] were used as a basis with the following exceptions: The total lime (CaO) content was not permitted to exceed 50 per cent, and the magnesia (MgO) not to exceed 23 per cent. The tensile strength tests on neat specimens were waived, but we required a compressive strength test on (1:2) standard Ottawa sand mortar 2-in. cubes, in which 25 per cent substitution of natural cement was made, to develop at least 70 per cent of the strength obtained with the same mortar in which no substitution was made.

Additional laboratory tests not called for in the specifications were made to determine the resistance to sulfate action, autoclave expansion tests and volume change measurements.

In general, it was found when either natural cement G or H was blended with the same non-sulfate resistant portland cement, that the blends became more resistant to the action of sulfate solutions.

Autoclave expansion tests showed the greatest variation between the two types of natural cement. Natural cement G, which was manufactured in vertical kilns, showed over 10 per cent expansion in the autoclave test, whereas natural cement H, made in a rotary kiln had only 0.3 per cent expansion.

On the basis of the physical tests on laboratory fabricated specimens with natural cement or their blends with the same portland, it was impossible to determine the characteristic differences evident when the cores taken from the pavements were subjected to chloride freezing-and-thawing tests. Briefly summarized, it was found after testing several hundred concrete cores removed from the 13 experimental projects and many others constructed throughout New York State, that portland cements of varying composition when blended with natural cement G were substantially superior in chloride resistance to the blends with natural cement H. Details concerning the data on these tests will be found in the 1938 *Proceedings* of the American Association of Highway Officials of the North Atlantic States.[3]

In order to differentiate between the properties of the two types of natural cement, an investigation was made to determine their chemical activity. A study to find out whether natural cements were puzzolans revealed to me that they were not, according to the definition of that term. In fact there was no fixation of lime by either of the natural cements when they were tested in a saturated lime water solution. Instead, there was an increase in the lime saturation of the solution.

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[1] Associate Laboratory Engineer, New York State Department of Public Works, Division of Engineering, Albany, N. Y.

[2] 1926 Book of A.S.T.M. Standards, Part II, p. 7.

[3] Ira Paul, "Chloride Salts-Resistant Concrete in Pavements," *Fourteenth Annual Proceedings*, Am. Assn. Highway Officials of the North Atlantic States, pp. 144–167 (1938).

The natural cements were then subjected to a "hydration test," which is a modification of my "water test" on portland cements. In the hydration test, the aqueous solution of the natural cement was analyzed for lime (CaO) content at the 7-day and 28-day periods. The lime liberated with natural cement G was found to be less than 2.5 per cent in 7 days, and not more than 3.0 per cent at the end of 28 days. With natural cement H the lime release ranged between 3 and 4 per cent at 7 days, and between 4 and 8 per cent in 28 days. In other words, by placing the proper limits on the 7- and 28-day hydration test, I was able to differentiate between the good and poor natural cement.

In our present specifications for natural cement we have incorporated this test method limiting the lime release not to exceed 2.5 per cent in 7 days and 3.0 per cent in 28 days. We have eliminated the mortar briquet tests because of the low range of strengths, but are requiring the neat briquet specimens to pass a 7-day strength test of at least 125 lb. and 200 lb. in 28 days.

About four years ago we experimented on two other types of admixtures with portland cement. In one project we added finely pulverized magnesium silicate to the concrete mix in the proportion of 50 lb. per cu. yd. of concrete. In the second project amorphous colloidal silica was added to the mix in the proportion of 4 lb. per sack of portland cement. The purpose for adding these admixtures was to increase the workability of the concrete, plus the puzzolanic property claimed for the colloidal silica. The chloride freezing-and-thawing tests made on the pavement cores with the two different admixtures showed no benefit over that of the straight portland and high-early-strength cements with no admixture on the same projects.

In conclusion I wish to caution the user of prepared blended cements not to be fooled by high-pressure sales talk that their product can be used as a "cure-all" for some of the ills and ailments of portland cement concrete.

MR. F. H. JACKSON.[4]—While a great deal of publicity has been given the matter of scaling in New York State, I personally have seen considerable scaling on concrete pavements in many other northern states. This scaling is, I think, undoubtedly associated with freezing and thawing and is, I believe, primarily the result of water gain or segregation during placing. This results in the formation of a thin porous layer on the top of the pavement which is an easy prey to the action of ice and salts used for ice removal. It is my opinion that the principal effect of the natural cement is to reduce the amount of this water gain, thereby producing a more uniform mix from bottom to top of the slab and consequently a denser surface. I do not think there is anything very mysterious about it. Furthermore, there may be other materials that will accomplish the same thing. Also, greater attention to the gradation of aggregate—possibly more fines in the fine aggregate—would, I think, help. It does seem to be a fact that, with at least one of the natural cements investigated, we observed this beneficial effect not only on core specimens taken from certain experimental roads but also through observation of the present condition of many of the roads in New York State. On these roads scaling, so far as my own personal observation is concerned, seems to be more prevalent where the straight portland cement is used than where the blend is used.

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[4] Senior Engineer of Tests, U. S. Bureau of Public Roads, Washington, D. C.

MR. PAUL.—In connection with these cements, I want to state that we have had some good results with straight portland and high-early-strength cements originating from one particular mill, without incorporating natural cement in the concrete mix. Another portland cement mill in the same locality, drawing its raw materials from the same source, and to all intents and purposes supposedly manufactured in the same way, did not function in the same manner without the natural cement. There must have been some variations in the mill control during the manufacture of the cements which gave rise to such differences in properties.

Also, it was evident from the field examinations while the pavements were laid that in general the items 935 and 950 showed very little or no bleeding, whereas the other two items of the higher C_3A content did show bleeding, and that bleeding was eliminated when the natural cement was added.

A STUDY OF FINE AGGREGATE IN FRESHLY MIXED MORTARS AND CONCRETES

By C. A. G. Weymouth[1]

Synopsis

The ordinary laws of voids stated for dry aggregates do not apply to the voids of mixtures of aggregates with cement paste. It is necessary to study the structure of the total voids and differentiate between the voids due to the paste, to boundary effect at the surfaces of the aggregate particles, and to particle interference before a clear understanding may be had of the influence of grading in aggregates upon the cement paste and various characteristics of fresh mixtures.

It is shown that the mortar-voids chart with certain mathematical curves added to it is valuable for the study of mortar sands and the blending of sands. The "boundary effect" and voids of "particle interference" can be measured, sands may be compared and poor gradings may be distinguished from excellent gradings by means of the mortar-voids chart.

Practical use is made of the mortar-voids curve to determine the water-cement ratio of a concrete mixture of desired relative consistency, when the mixture has no particle interference due to the coarse aggregate.

The importance of the characteristics of the fine aggregate to the properties of concrete was recognized at an early date. Feret (**1**),[2] one of the first to conduct comprehensive investigations of portland cement mixtures, studied the relationship of the granulometric composition of sand to the voids, porosity, strength and other characteristics of mortar. Talbot and Richart (**2**), in their classical study of concrete, devoted major attention to the mortar and demonstrated the voids in the mortar to be a principal criterion of concrete quality. Fuller (**3**), who advanced an elliptical curve as an "ideal" grading for sand; Edwards (**4**), who evaluated the grading of aggregates on the basis of their surface areas; Abrams (**5**), who developed the fineness modulus theory; and many other students of concrete contributed greatly to our knowledge of the effect of aggregate gradation on the characteristics of concrete (**6**,**7**).

However, none of this earlier work developed information permitting of a detailed examination of the effects of particle distribution in fine aggregate, nor did it take into account, in an adequate manner, the effect of the gradation of the cement. The Talbot-Richart procedure measures an *effect of particle distribution* but does not evaluate it in terms of grading. Surface areas and fineness moduli are functions of an

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[1] Concrete Engineer, The Raymond G. Osborne Laboratories, Los Angeles, Calif.

[2] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 372.

"average" of the grading and many major variations in the arrangement of the particles are not thereby detected.

The author has developed a procedure for the examination of mortar mixtures which permits of studying in detail the effect of the grading of the aggregate and which even affords some information on the "grading" of the cement. It uses as a basis the mortar-voids relationship developed by Talbot and Richart and divides the voids into three classifications: those originating in the cement paste, those caused by "boundary effect" due to the surface area of the aggregates, and those due to "particle interference" in the grading of the aggregate. The purpose of this paper is to show the application of that procedure to studies of fine aggregate in freshly mixed mortars and concrete.

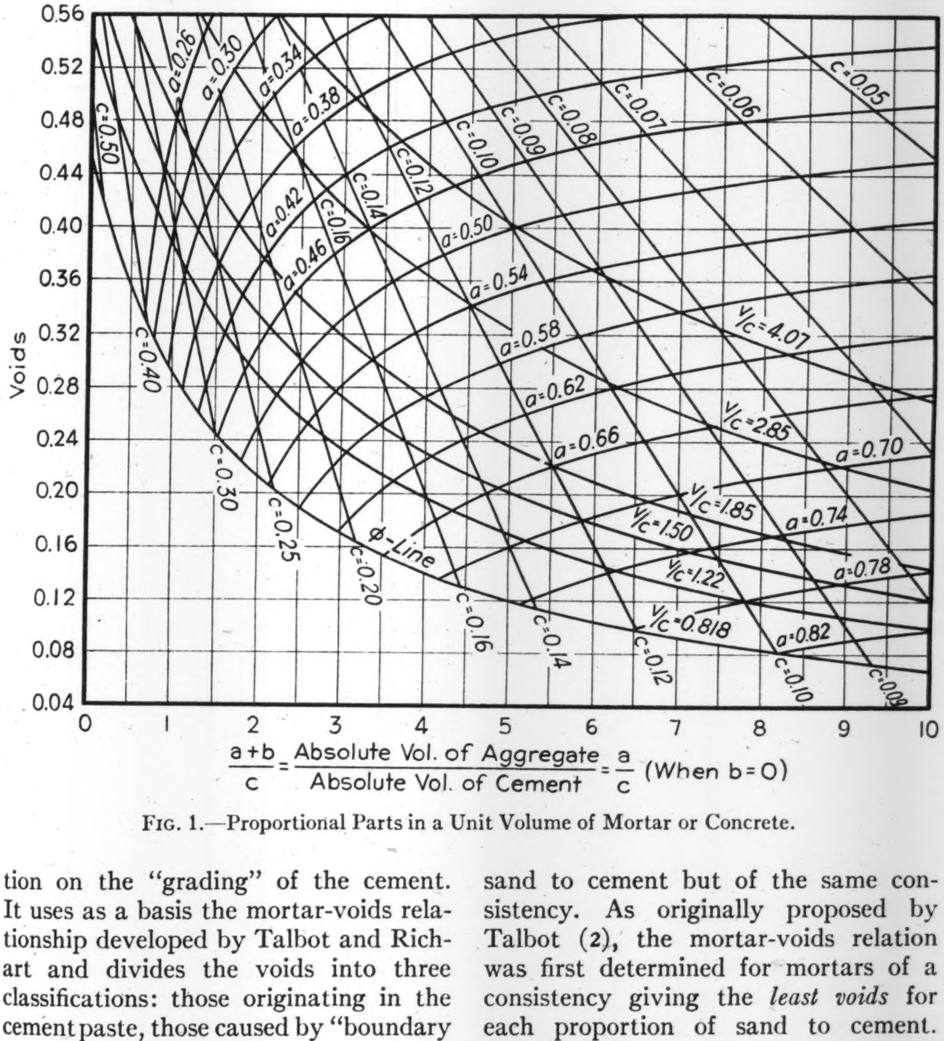

FIG. 1.—Proportional Parts in a Unit Volume of Mortar or Concrete.

THE MORTAR-VOIDS RELATIONSHIP

The mortar-voids relationship for a sand is established by measuring the voids in mixtures of differing ratios of sand to cement but of the same consistency. As originally proposed by Talbot (2), the mortar-voids relation was first determined for mortars of a consistency giving the *least voids* for each proportion of sand to cement. The "basic water content" was carefully determined for each mix. When curves for wetter mortars were plotted, the water used was taken as a fixed multiple of the basic water content, and called the "relative water content." But the same relative water content does not give the same relative consistency, or slump, with different sands. The present author accordingly uses a consistency that gives a 6-in. slump to the concrete—chosen because it is usual in architectural concrete. This corresponds to a $3\frac{1}{2}$-in. flow with ten 1-in. drops in a 3-in. Burmister flow trough (**8**). (All dimensions are one-half those of the standard 6-in trough.) The water-cement ratios for other slumps may be interpolated from the consistency chart, explained later, or a different consistency may be chosen for the mortar-voids curve.

Mathematical Reference Curves:

All changes in mortars, such as changing from one sand to another, or to a different cement, changes in the grading, water-cement ratio, etc., affect the volume of the voids in the mortar. The significance of these effects, and others, is made clear by comparing the position of the point, representing the mortar tested, to a series of fixed reference curves.

Talbot and Richart's basic equation is:

$$v + c + a + b = 1 \ldots\ldots (1)$$

where v is the volume of the voids, and c, a, and b are the absolute volumes of the cement, sand, and coarse aggregate, respectively, in a unit volume of concrete.

The mortar contained in a unit volume of concrete equals $(1 - b)$, and the voids in a unit volume of the contained mortar are:

$$v_m = \frac{v}{(1 - b)} \ldots\ldots\ldots (2)$$

It is convenient to use v_f in Eq. 2 in place of v_m when the voids referred to are those in a unit volume of the material finer than that represented by b.

When mortars are being studied separately, Eq. 1 is written:

$$v + c + a = 1 \ldots\ldots\ldots (3)$$

Three series of reference curves are obtained from Eq. 1 by putting $(a + b)/c = k$, or (for mortars, where $b = 0$) from Eq. 3 by putting $a/c = k$, upon which we may write:

$$c = \frac{1 - v}{1 + k} \ldots\ldots\ldots (4a)$$

$$a = \frac{k(1 - v)}{1 + k} \text{ from Eq. 3, or}$$

$$(a + b) = \frac{k(1 - v)}{1 + k} \text{ from Eq. 1} \ldots (4b)$$

$$\frac{v}{c} = \frac{v(1 + k)}{1 - v} \ldots\ldots\ldots (4c)$$

These mathematical values for the cement content, aggregate content, and voids-cement ratio apply to all parts of the mortar-voids chart, and are not dependent on any particular sand or aggregate.

Equation 4 (a) gives a series of straight lines for values of c as plotted in Fig. 1 radiating from a point well above the left-hand corner. Equation 4 (b) gives a series of curves for values of a, running from the lower left-hand corner to the upper right. Equation 4 (c) gives a series of curves for values of v/c, running from the upper left-hand corner, where they are almost vertical, to the lower right-hand corner, where they are almost horizontal.[3] Any point interpolated from these curves in Fig. 1 satisfies Eqs. 1 and 3.

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[3] F. E. Richart gives equations for the last two series of curves with somewhat different form and for a different purpose, in a discussion of analytical properties of mortars appearing in *Proceedings*, Am. Soc. Testing Mats., Vol. 25, Part II, p. 295 (1925).

CONSISTENCY OF MORTARS AND CONCRETES

The consistency of mortars and concretes depends upon the flow properties of their cement pastes. The flow of a given neat cement paste varies directly with the amount of water contained; when aggregates are added much larger water contents are required to give flows equivalent to the flow of the original paste. Relative consistency, as used in this paper, equates the consistency of a plastic mortar to that of neat cement paste when the interior structure of the cement paste (defined by the spacing of the cement particles and by the amount of water and air associated with each cement grain) is the same in the mortar and neat cement paste.

Flow of mortar or concrete is caused by the shearing of layers of cement paste, and the structure of the paste determines the ease of flow because it determines the ease of shear. This shear does not involve the cutting of the solid grains of matter but occurs in the water films between the grains of cement. The effect of flow is continually to remold the stream of material. Large particles are carried along in suspension in this stream and it is the structure of the cement paste that provides this property of suspension. At any particular instant in the flowing stream, this structure is disturbed at the planes of shear, but in other portions of the mass it is definite. As the flow proceeds, these planes of shear constantly change with the remolding of the mixture.

The usual apparatus for measuring relative consistency, such as the standard slump cone, the flow table, or the flow trough, involves the application of momentary force to secure this shear and consequent flow. All these three produce the same kind of effect on the material tested and results should, therefore, bear a simple relation to one another. If they do not, it is proof that there are elements in the mixture which interfere with the flow of the material in the manner described. The author's experiments indicate a simple relationship between the slump cone and the flow trough. The correlation of the three methods of test, however, holds good only for those mixtures in which the cement paste dominates. Those mixtures in which the aggregates dominate by their close spacing are not true suspensions of aggregate in cement paste, and flow is not wholly governed by the structure of the cement paste. Hence tests for consistency on mixtures at the border line of harshness with these three methods do not, necessarily, give equivalent results.

When saturated, surface-dry sand is added to cement paste, the mixture will be stiffened. Each grain of sand, even when separately spaced and independent of the mass of grains, extracts water from between the cement particles to satisfy a condition existing at its surface when surrounded by cement paste. In a paste of wet consistency this subtraction of water draws the cement particles closer together, and hence the mixture is stiffened because of the increased friction on the water films between the cement grains. If the paste is at basic water content, that of densest mixture, the loss of water to the sand grains will result in a change from a plastic to a granular condition in which air takes the place of water and increases the spacing of the cement grains.

Adding just enough water to replace that extracted by the sand restores the internal structure of the paste and its consistency, and the mortar is now said to have the same relative consistency as the original cement paste. This additional water is dependent upon the surface areas of the sand grains. It is proportional to the amount of sand added and tests show it to be much in excess of that required to form wetting films on the sand grains. This greater amount is associated with an increase in the voids in the cement paste at the surfaces of the sand grains due to what the author terms "boundary effect."

Up to this point it has been assumed that the sand grains were widely separated from one another. If more sand is added so that the grains are crowded together beyond a critical amount there will be a further increase in the voids of the cement paste brought about by "particle interference." Those voids likewise require more water and air in the mix to maintain the relative consistency, and this makes for a further increase in the water-cement ratio.

TABLE I.—VALUES FOR 6-IN. SLUMPS INTERPOLATED FROM SERIES 186 OF THE PORTLAND CEMENT ASSOCIATION.

Mix	a/c (Eq. 1)	b/c (Eq. 1)	Water-Cement Ratio
1:1 :0	1.413	0.000	0.54
1:1 :1	1.413	1.357	0.54
1:1 :2	1.413	2.712	0.56^a
1:1 :3	1.413	4.08	0.64^a
1:1 :4	1.413	5.43	0.77^a
1:1½:0	2.118	0.000	0.59
1:1½:1	2.118	1.357	0.61
1:1½:2	2.118	2.712	0.63^a
1:1½:3	2.118	4.08	0.70^a
1:1½:4	2.118	5.43	0.80^a
1:1½:5	2.118	6.79	0.90^a
1:2 :0	2.825	0.000	0.69
1:2 :1	2.825	1.357	0.71
1:2 :2	2.825	2.712	0.74
1:2 :3	2.825	4.08	0.76^a
1:2 :4	2.825	5.43	0.80^a
1:2 :5	2.825	6.79	1.06^a
1:2½:0	3.530	0.000	0.81
1:2½:1	3.530	1.357	0.87
1:2½:2	3.530	2.712	0.87
1:2½:3	3.530	4.08	0.93^a
1:2½:4	3.530	5.43	1.02^a
1:2½:5	3.530	6.79	1.16^a

[a] Particle interference due to coarse aggregate.

Relation of Consistency of Mortar and Concrete:

In designing concrete mixtures from a mortar-voids curve of a sand, made with mortars of the same relative consistency, it is implied that the slump of concrete is identical with, or proportional to, that of the mortar it contains. The author finds that this is true *for concretes in which there is no particle interference in the coarse aggregate* and which is sufficiently cohesive to suspend the heaviest sizes. Table I tends to confirm this with data interpolated from tests by the Portland Cement Association. Even with the poorly graded sand used (only 7 per cent passing the No. 48 sieve) the water-cement ratio in each family of concretes is nearly constant in the mortar and concretes until particle interference causes a sharp increase in it. In the leaner mortars the suspension properties are poor and the correlation is not so evident.

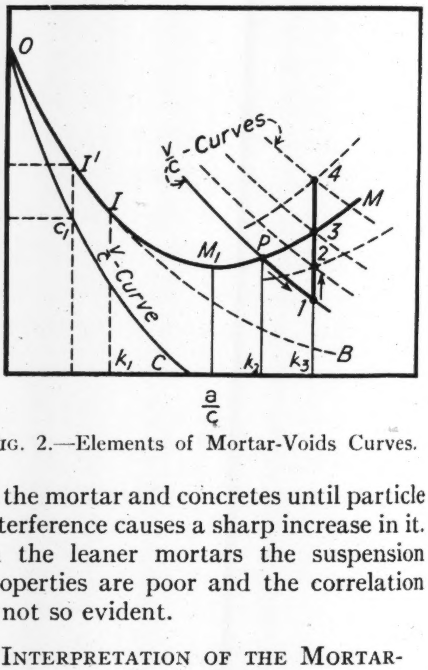

FIG. 2.—Elements of Mortar-Voids Curves.

INTERPRETATION OF THE MORTAR-VOIDS CURVE

In Fig. 2, curve *OIM* is a typical mortar-voids curve, each point representing a mortar made from the same sand and cement but of differing proportions, mixed to the same relative consistency. The average compaction of the cement particles in the neat cement paste at *O*, on the left ordinate, is expressed by the cement-space ratio,

$\frac{c}{v+c}$, which is related inversely to the voids-cement ratio, v/c. The voids-cement ratio curve, OC, derived from Eq. 4(c), represents the degree of compaction of the neat cement paste and serves as a reference curve with which to compare the voids in any mortar with the voids in the neat cement paste of like consistency. It can be shown by test data that when only large particles are added to the paste, the mortars will give points close to the curve, OC; but with fine sand the points are much higher.

For a mortar at I', the original voids in the cement paste contained in the mortar are shown by the height of C_1 and the voids introduced into the mixture by the sand are equal to $I'C_1$. These additional voids must be taken as a part of the cement paste. It is therefore evident that the structure of cement paste in mortars is different from that in neat cement paste. The interior particles of the paste mass have the same spacing as the neat cement paste of like consistency, but the particles adjacent to the sand grains contain a higher percentage of voids. To the left of a critical point, I (Fig. 2), the rise in voids from C_1 to I' is due to boundary effect alone; the sand grains are widely spaced and the mortar is dominated by the plasticity of the cement paste. To the right of this point the rise in voids is due to both boundary effect and particle interference, and the mortars are dominated by the fine aggregate. The curve OIB represents the position the mortar-voids curve would have if there were no particle interference to the right of I, and is called the boundary-effect curve.

Figure 2 illustrates the three kinds of voids in a mortar: those in the original cement paste, measured by the height of the curve for the neat cement paste OC; those due to boundary effect, measured by the vertical distance from OC to the boundary-effect curve OIB; and those due to particle interference, measured by the vertical distance from curve OIB to the mortar-voids curve (OIM).

Boundary Effect:

As the term is used by the author, boundary effect is the difference in the percentage of voids at any bounding surface and the percentage of voids within a mass of particles, all the particles being of the same size. This is illustrated in Fig. 3. The lower layer of spheres forms boundary voids with the top of the table, and void pockets with the layer above. It is evident that the voids formed between the upper and lower layers are smaller than the voids formed between the spheres and the table top, because the upper layer of spheres sinks down into the lower layer, below the line T-T. The boundary voids are formed on every surface, including the surfaces of all grains larger than the smallest grains. Only the finest grains form boundary voids unless there are not enough of them for all the exposed surface, in which case the next larger grains do also. With cement-sand mixtures, boundary voids are formed by cement-flour particles.

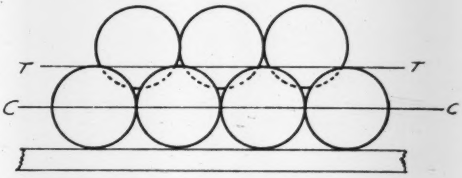

FIG. 3.—Boundary Voids.

The percentage of voids at the boundary surfaces depends upon the depth of the boundary voids and this depends upon the size of the particles forming them. This average depth also depends upon the consistency of the cement paste, since all void spaces in plastic mortars increase in volume with additional water. With these conditions constant, boundary effect in mortars is proportional to the surface area of the sand, or (with a different factor) to the absolute volume of the sand, in the mortar.

In Fig. 2, for a point I' to the left of I, the boundary effect is equal to $I'C_1$, or $v - v_c$. With the above conditions constant, we have

$$\text{Boundary effect} = v - v_c = e\,a \quad \ldots (5)$$

where e is constant. Let v/c in the curve OC_1C, be represented by j, a constant, then by Eq. 4(c), $v_c = j/(1 + k + j)$. Then with Eq. 4(b), and substituting for a in terms of k and v, we can write:

$$e = \frac{1 + k}{k(1 - v)}\left(v - \frac{j}{1 + k + j}\right) \quad . . (6)$$

Equation 6 provides a means of locating the point I at which particle interference begins, since to the right of this point, as has been explained in discussing Fig. 2, the rise in voids above the voids-cement reference curve is caused by voids of both boundary effect and particle interference and is therefore no longer proportional to the surface area of the sand. Thus the value of e must become variable to the right of I. If the cement particles just larger than the finest flour must be called into play to form boundary voids for large values of a/c, e will increase to correspond, but will be constant at this new value until particle interference causes a variable increase in e.

Particle Interference:

In Fig. 4(*a*) is shown a mass of small particles of the same size into which a few large particles have been introduced. The average distance, t, between the large particles is much greater than the diameter, D_1, of the smaller particles. It is evident that the average size of the individual void in the mass of small particles is not particularly effected by a few large particles, and that the small particles are in point to point contact throughout.

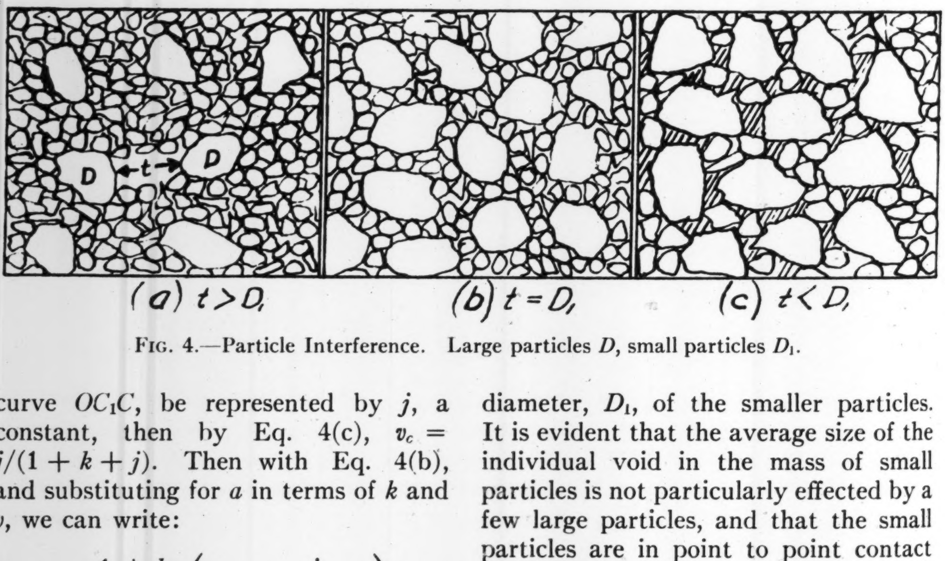

FIG. 4.—Particle Interference. Large particles D, small particles D_1.

In Fig. 4(*b*) the number of large particles has been increased until the average clear distance, t, between them is just equal to the average diameter of the smaller particles. The smaller particles still have continuous point-to-point contact, and the average size of individual void spaces contained in them is unchanged, although there is an increased boundary effect with the addition of more of the larger sizes. This proportion of large and small grains in a mixture of the two is critical, as any further addition of large grains will seriously affect the structure, destroying the point-to-point contact of the small grains and "bunching" them with new voids between the bunches as is shown in Fig. 4(*c*). Reducing the clear distance between the large particles until it is less than the diameter of the small particles makes it impossible for these particles to fill homogeneously the large voids which are shown hatched in the diagram.

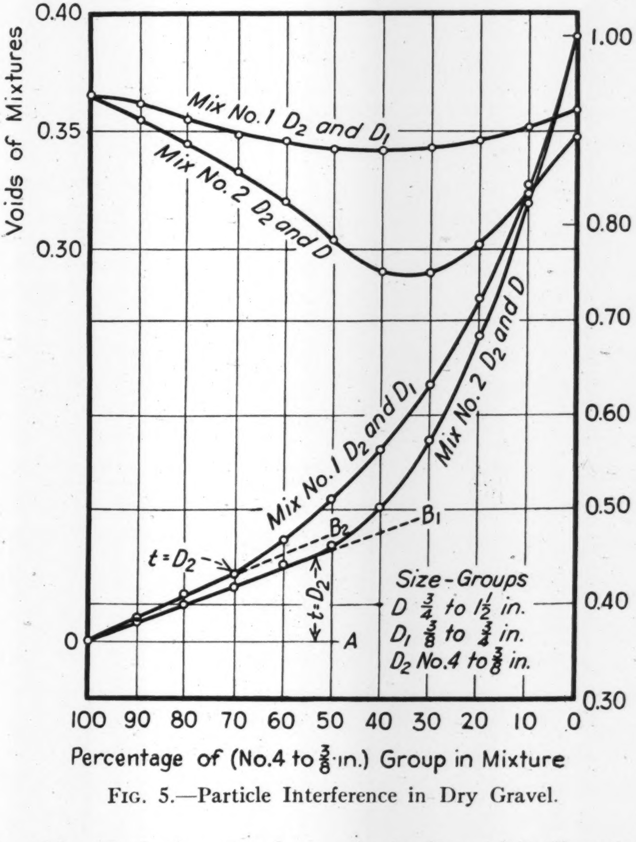

FIG. 5.—Particle Interference in Dry Gravel.

The fact that the critical point where particle interference begins is where the average clear distance between the large particles equals the average diameter of the small particles is further brought out by Fig. 5, the upper curves of which have been plotted from data interpolated from Stanton Walker's diagram on the voids in gravels (9). The lower curves show the voids referred to the smaller particles, computed by $v_f = v/(1 - b)$. Each curve starting from *O* shows a gradual straight-line increase in the voids to a point where the curve takes a sharp upward trend. This point is where the clear distance, *t*, between the large particles has been reduced to the average diameter of the small particles. The boundary effects are measured by the vertical distance from *OA* to OB_1 and OB_2, respectively, and particle interference is measured from the lines OB_1 and OB_2 to the respective curves above.

The formula (10, 11) derived by the author from which the average clear distance between the larger particles is calculated is:

$$t = \left[\left(\frac{d_o}{d_a}\right)^{\frac{1}{3}} - 1\right] D$$

in which d_o is the dry-rodded density (solid volume per unit bulk volume) of the particles of the size-group[4]; d_a is the relative density of the same particles as they are spaced in the mixture (or the concentration taken relative to the space in which the particles are distributed, which is the bulk volume of the mixture minus the absolute volumes of all particles having larger diameters), D is the average diameter of the particles of the given size-group,[4] and t is the average clear distance between them.

TABLE II.—VALUES OF d_o AND d_a CRITICAL FOR $t = D_1, D_2,$ OR D_3.

	Dry-Rodded Voids			Values of d_a		
				Kind of Grading		
					Groups in Gap	
	d_o	Decimal of Unit Volume	Per cent	Continuous $0.296d_o$	1 $0.512d_o$	2 $0.703d_o$
Dominant group				D	D	D
Next smaller diameter				D_1	D_2	D_3
Nearly round	0.650	0.350	35.0	0.192	0.333	0.457
Sub-angular	0.615	0.385	38.5	0.182	0.312	0.432
Broken, cubical	0.580	0.420	42.0	0.172	0.297	0.408
Broken, disk-like	0.550	0.450	45.0	0.163	0.282	0.387

Note.—In wet concrete mixtures, values for jump gradings should be reduced 20 per cent for coarse size-groups because of the tendency of heavy particles to sink and become more concentrated than in average mixture.

With d_o and D constant, d_a is the only variable in the equation, and it is therefore sufficient to find the value of d_a that will make t equal to D_1 and to use it as a criterion. For jump gradings, where one size-group is omitted, d_a may be such as to make t equal to D_2, the average diameter of the next smaller size group, and where two sizes are omitted t may equal D_3. The calculations for these are shown in Table II.

The values of d_o in Table II apply to size-groups with large as well as small diameters, since d_o is independent of the magnitude of the diameter. The effect of particle interference is always to increase the voids referred to the mixture of fine particles contained within the void pockets[5] of the group causing the interference. The ratio d_o/d_a, measures the dispersion, or separation, of the particles of this group. For workable mortars and concretes no size-group in the mixes should have values of d_a exceeding those of Table II.

TABLE III.—SANDS COVERED IN TALBOT'S STUDIES.

From Bulletin 137 (2).

	Fineness Modulus	Surface Modulus	Voids	Per Cent by Weight Retained Between Square-Mesh Sieves						
				−100	100 to 48	48 to 28	28 to 14	14 to 8	8 to 4	4 to 3/8
No. 3 rounded sand	2.79	22.6	0.309	5	14	27	21	17	16	
No. 6 limestone screenings	2.40	37.0	0.280	23	14	15	15	17	13	3
No. 18 angular sand	3.50	13.7	0.361	1	6	17	24	27	19	6
No. 23 rounded sand	1.47	39.8	0.382	6	42	51	1			
No. 36 subangular sand	3.46	10.2	0.378	0	0	3	60	25	12	

EFFECT OF GRADING

The study made by Talbot some years ago (2) covered sands, both natural and artificial, from widely scattered areas, and yielded a mass of data. Typical sands from that study are shown in Table III.

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[4] By size-group is meant that portion of the aggregate which is held between two successive sieves of any standard series, provided the wire openings have a constant ratio. In this paper the sieves used (Tyler) have a ratio of 2.

[5] The author uses the term *void pocket* to designate an individual void, surrounded by particles of the same size-group, whether or not this void contains smaller particles.

In Fig. 6 three sands from Table III are compared, a very fine sand graded from No. 200 to No. 28 sieves; a coarse sand graded from No. 28 to No. 4 sieves; and an approximate blend of the two, graded from No. 200 to No. 4 sieves. The mortar-voids curves intersect the zero ordinate at *O*, the point of neat cement paste of basic consistency. The fine sand mortars with sand No. 23 have the greatest voids throughout, those due to boundary effect being greater than those due to particle interference. The curve for the coarse sand, No. 36, lies closest to the v/c curve of reference up to the point where particle interference begins, and for the lean mortars beyond this point the voids are increased so much by particle interference that they are almost as much as those of the fine sand mortars. The curve for sand No. 3 lies between the others for small a/c values and below both for values of a/c above 3.00. This sand has the least particle interference in the leaner mortars. It will make the best concrete because it has, in addition to low voids, sufficient fines to make a mortar which will hold up the heavier pieces of the coarse aggregate.

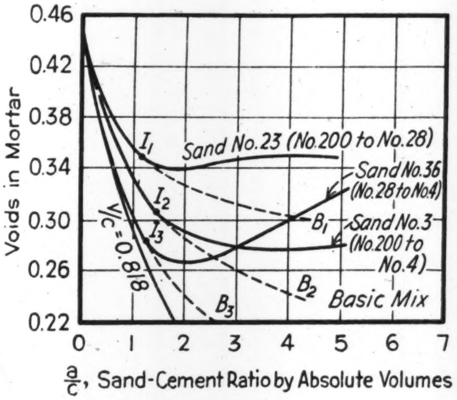

FIG. 6.—Effect of Grading Limits.
Well-graded sands with rounded grains.

Sand No. 6, limestone screenings (see Fig. 7), has 37 per cent of its particles finer than the No. 48 sieve, while sand No. 18, natural angular grains, has only 7 per cent of its particles finer. The first with greater surface area has practically no particle interference throughout the curve, which is nearly identical with its boundary-effect curve. This sand would be improved by reducing the amount of fines. The second sand has little boundary effect but has large particle interference beyond $a/c = 1.50$, and, like sand No. 36, would give poor support in concrete to the coarse aggregate.

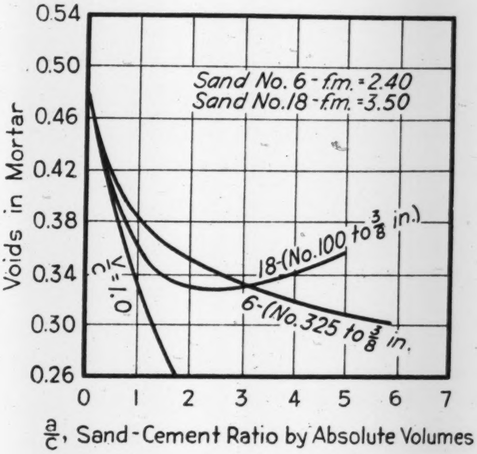

FIG. 7.—Effect of Fines (200–50).
Very angular sands 1.20 relative water content.

EFFECT OF SHAPES OF PARTICLES

Angular grains, tested with the usual sieves with square openings, give a coarser product in any size-group than round grains, because they pass upended and diagonally. The position of the mortar-voids curve is influenced by the surface area, frictional effects due to shape, and volume per grain. It can be shown that the surface areas of round and angular particles of a *given absolute volume* of sand in a size-group are practically the same; hence the effect of greater volume per grain in a size-group is neutralized by this equality of surface area. The important difference in sands of spherical and angular shapes is the frictional effect which requires more water for the same degree of compaction, with the result that mortar-voids curves of angular sand, other things being equal, stand higher than those of rounded sand. There is a difference even between rounded grains; the well-rounded grains of Elgin sand give higher curves than the smoother spherical Ottawa sand, both screened to the standard size.

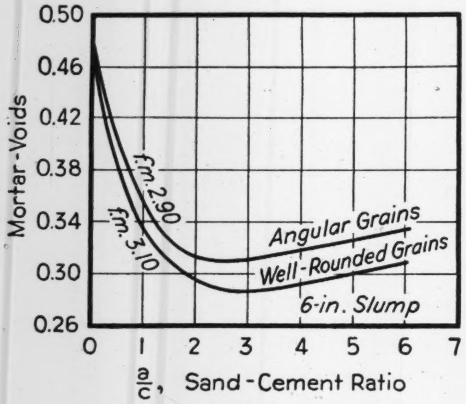

FIG. 8.—Effect of Shape of Particles.

Angular-grain sands should have a larger proportion of material passing the No. 48 sieve to hold the mixing water. To be comparable, angular sands should have a lower fineness modulus than rounded ones. Sands made by crushing stone especially need additional fines. Figure 8 shows the difference between mortars made with rounded grains and angular grains of nearly equivalent sieve analysis.

THE CONSISTENCY CHART

Mortars may be classified according to their air contents as overdry mixes, plastic mixes, and overwet mixes. In the first the cement is "bulked" like moist sand, the mortars are friable, and when hardened have low strength. With basic water content the air is reduced to a critical amount, which gives minimum voids, and the mortars become plastic and capable of a slight slump. With further decrease in air, accomplished by increasing the water content the slump increases. At the point of zero air, or inundation, plastic cohesion disappears. These changes are shown in Fig. 9.

This air content, expressed as the ratio of its volume, at ordinary mixing temperature, to the volume of the mixing water, is seen to be structural and not accidentally entrained when it is found to be identical in oft-repeated tests with a given cement and with the same proportions of mix and water. The author has found from his studies that the value of this air ratio in cement pastes, mortars and concretes has as much significance to consistency of mix as the water-cement ratio has to strength.

If A and w are the volumes of air and water, respectively, in a unit volume of mixture, the voids are, $v = A + w = (1 + A/w)w$. By putting $Z = A/w$, the voids can be written,

$$v = (1 + Z)w$$

and the voids-cement ratio as:

$$v/c = (1 + Z)w/c \ldots\ldots (7)$$

(The ratio w/c is the water-cement ratio by absolute volumes. Multiplying by 0.480 gives the water-cement ratio by bulk volumes, and by 3.59 gives gallons per sack of cement.)

The relation between the voids-cement and water-cement ratios given in Eq. 7

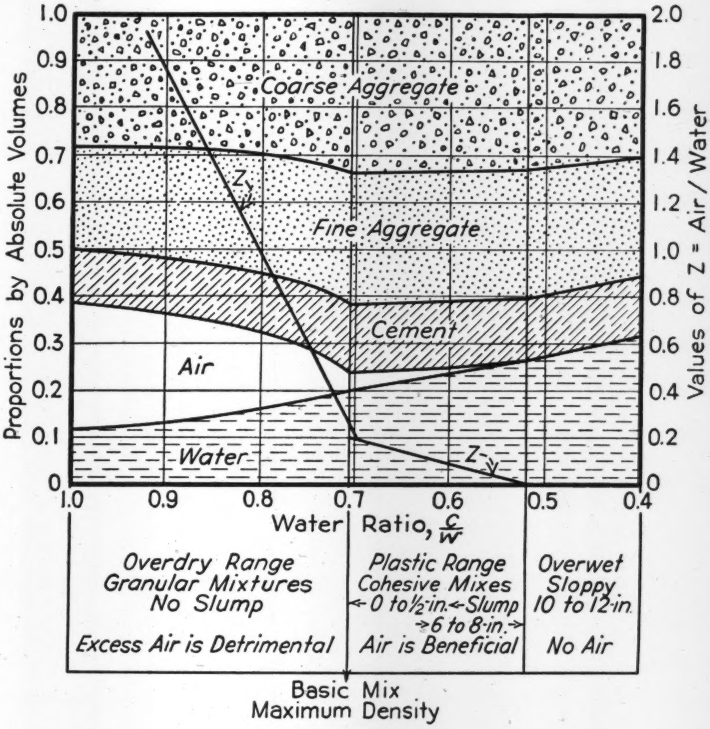

FIG. 9.—Structural Air in Concrete.

Data from report by W. A. Blanchette in *Public Roads*, May, 1934, Table 3: $a/c = 2.00$ $b_s = 0.45$.

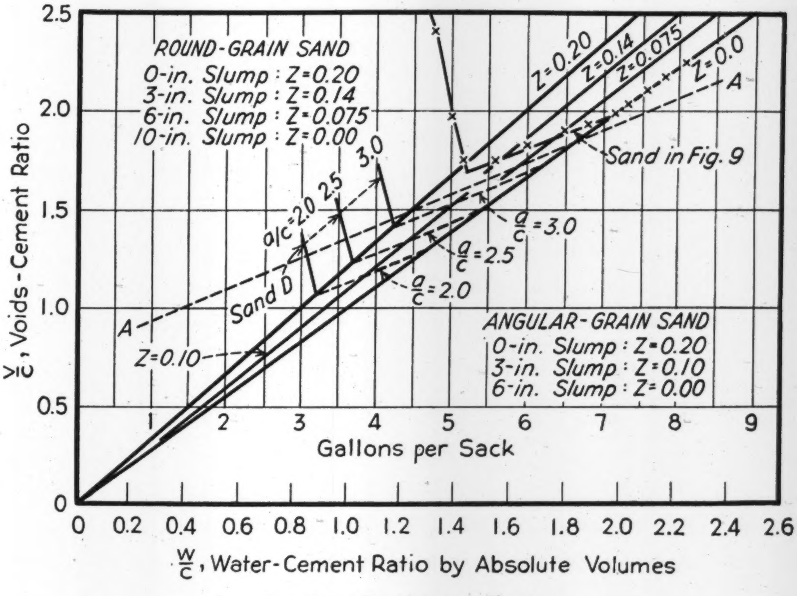

FIG. 10.—Consistency Chart.

comprises the *consistency chart* shown in Fig. 10. The positions of the Z-lines are independent of test data but points from any series of tests may be plotted on the chart after computing the v/c and w/c ratios. The mixture illustrated in Fig. 9 is represented by crosses. The two points above $Z = 0.20$ are from the overdry consistencies; the others are in the plastic range falling on a line parallel to A-A. This line contains all the points in the plastic range for mixtures of this sand and cement at a fixed a/c value. Mixtures with other values of a/c give similar lines parallel, or nearly so, to A-A for variable water content. The slope of A-A seems to depend upon the fineness of the cement and upon the amount of surface in the sand to be covered by the cement flour, and the author's own experiments and examination of other experimental data bear this out. The line for neat cement paste is steeper than for mortars with the same cement, while mortars with large sand-cement ratios and surface areas or with finer sands (having larger surface areas) give flatter slopes to the A-A lines. The A-A lines for coarse sands fall lower on the chart than for fine sand for a given a/c value. The three lower lines drawn parallel to A-A in Fig. 10 are for a coarse sand discussed later.

TABLE IV.—RELATION OF v/c AND WATER-CEMENT RATIO FOR 6-IN. SLUMP.

Water, gal. per sack	Water-Cement Ratio, w/c	Voids-Cement Ratio, v/c	
		Angular Grains $Z = 0$	Round Grains $Z = 0.075$
4.0	1.114	1.114	1.198
5.0	1.393	1.393	1.497
6.0	1.672	1.672	1.797
7.0	1.950	1.950	2.096
8.0	2.228	2.228	2.395
9.0	2.506	2.506	2.694
10.0	2.786	2.786	2.995

Sands with angular grains require a larger proportion of the mortar voids to be filled with water than those with rounded grains for the same slump. If just enough water is added to concrete to reduce Z to zero, angular-grain sands give a 6-in. slump and round-grain sands a 10-in. slump. The slumps for any a/c mix vary from zero slump to a higher slump inversely with the value of Z, as is indicated in Fig. 10. This has been confirmed with data examined by the author from numerous series of tests.

The consistency chart shows the relation between the water-cement ratio and the voids-cement ratio for a uniform relative consistency, such as the 6-in. slump, and is of immense practical value in giving the water-cement ratio needed for any concrete free from particle interference in the coarse aggregate.

Table IV is obtained by interpolating from the consistency chart or is computed by Eq. 7. The same values of gallons per sack and of v/c are found on the line $Z = 0$ for the angular sands and on the line $Z = 0.075$ for round-grain sands, for 6-in. slumps. Curves for each of these v/c values are then plotted on a mortar-voids chart similar to Fig. 1. Instead of marking these with numerical values of v/c, the water-cement ratios in gallons per sack are indicated. Figure 11 shows such a relationship for round-grain sands and Fig. 12 for angular-grain sands—both figures representing mortars of 6-in. slump. Thus if it is desired to have a 6-in. slump with sand D of Fig. 12 using 6.5 gal. per sack, the mortar mix must have $a/c = 3.17$. Or, if the sand-cement ratio is given, the water-cement ratio needed can be read directly.

BLENDING OF SANDS IN PRACTICE

Three typical sands, with grains sharp and angular, are listed in Part 1 of

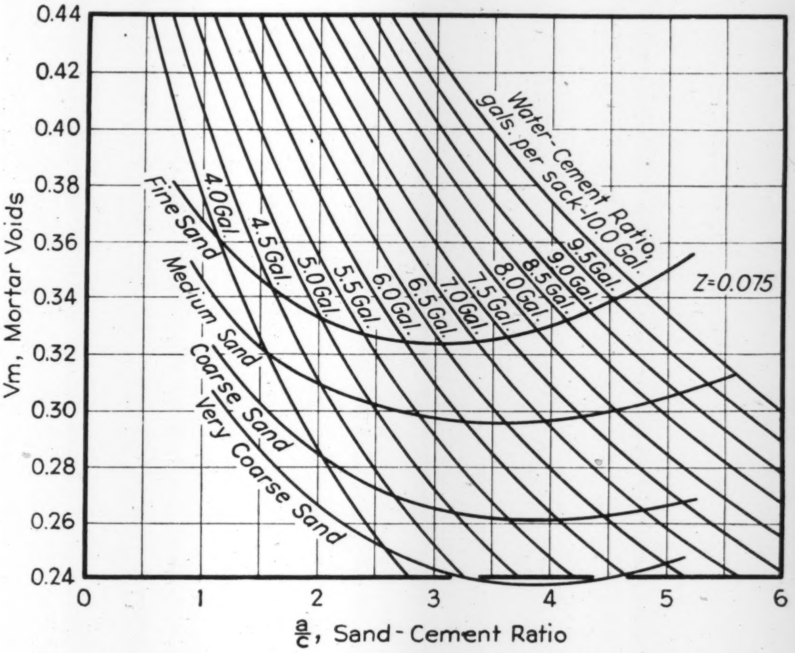

FIG. 11.—Mortar-Voids Curves Chart for Sands with Rounded Grains.
6-in. slump.

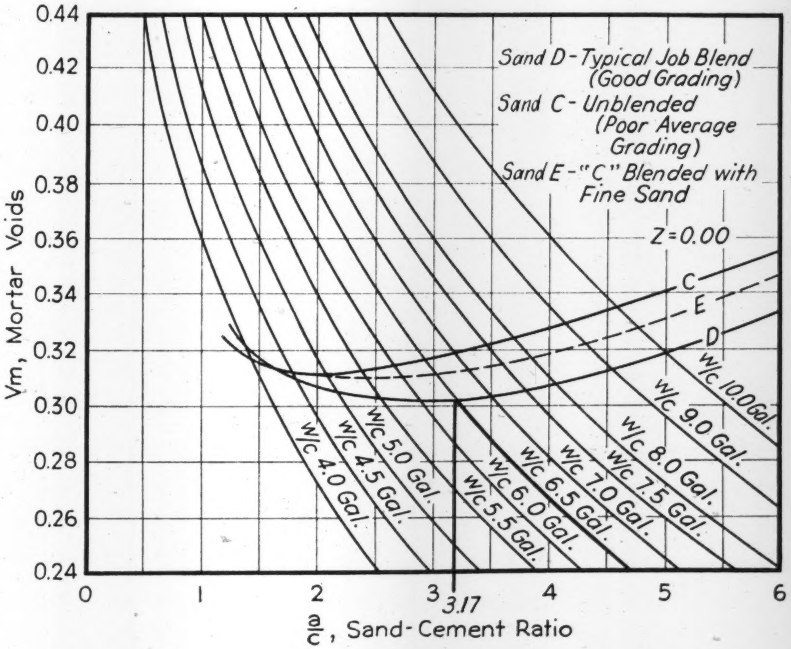

FIG. 12.—Mortar-Voids Curve Chart for Sands with Angular Grains.
Gallons per sack for 6-in. slump.

Table V. Sand A is a fine blending sand and sands B and C are concrete sands with little change in grading from month to month, too coarse for good concrete. Local practice with these angular sands gives best results with from 18 to 22 per cent passing the No. 48 sieve. The gradings marked D and E are blends of sands B and C, respectively, with sand A as shown. The mortar-voids curves of sands C, E, and D are given in Fig. 12. It will be seen that sand D requires the least water-cement ratio for a given value of *a*/*c*.

TABLE V.—EFFECT OF BLENDING SANDS.

	Fineness Modulus	− 200 Sieve	No. 200 to No. 100 Sieve	No. 100 to No. 48 Sieve	No. 48 to No. 28 Sieve	No. 28 to No. 14 Sieve	No. 14 to No. 8 Sieve	No. 8 to No. 4 Sieve	No. 4 to No. 3 Sieve
PART 1—GRADING OF SANDS: PERCENTAGE IN SIZE-GROUPS									
Sand A—Fine sand	1.15	18.2	19.4	32.5	16.2	6.2	5.7	1.8	...
Sand B—Roscoe sand	3.04	....	2.3	11.9	22.0	26.5	20.8	13.6	2.9
Sand C—San Gabriel sand	2.95	....	2.5	12.0	28.0	24.5	16.4	12.2	4.4
Sand D—88 per cent sand B with 12 per cent sand A	2.81	2.3	4.3	14.4	21.3	24.0	19.0	12.2	2.5
Sand E—88 per cent sand C with 12 per cent sand A	2.74	2.3	4.5	14.5	26.5	22.2	15.1	11.0	3.9
PART 2—COMPOSITION OF RELATIVE DENSITIES IN MORTARS, $a/c = 3.17$									
Sand D, $a = 0.530$		0.012	0.023	0.076	0.113	0.127	0.101	0.065	0.013
Subspace		0.482	0.505	0.581	0.694	0.821	0.922	0.987	1.000
Relative density, d_a		0.025	0.046	0.131	*0.163*	0.155	0.110	0.066	0.013
PART 3—COMPARISON OF RELATIVE DENSITIES FOR MORTARS, $a/c = 3.17$									
Sand D, $a = 0.530$, d_a =		0.025	0.046	0.131	*0.163*	0.155	0.110	0.066	0.013
Sand E, $a = 0.523$, d_a =		0.025	0.047	0.129	*0.190*	0.133	0.036	0.059	0.020
Sand C, $a = 0.519$, d_a =		0.000	0.026	0.111	*0.208*	0.153	0.093	0.065	0.023

PART 4—COMPOSITION OF MORTARS, $a/c = 3.17$

	Sand D			Sand E			Sand C		
	lb.	vol.	unit	lb.	vol.	unit	lb.	vol.	unit
Cement	94	0.480	0.167	94	0.480	0.165	94	0.480	0.163
Sand	245	1.522	0.530	245	1.522	0.523	245	1.522	0.519
Water	6.5	0.870	0.303	6.8	0.908	0.312	7.0	0.934	0.318
Volume, cu. ft.		2.872	1.000		2.910	1.000		2.936	1.000

Let us compare these sands mixed with cement to form mortars with *a*/*c* of 3.17 and a 6-in. slump. The composition of these mortars is given in Part 4 of Table V. The absolute volume of a sack of cement is taken as 0.480 cu. ft. and the weight of a cubic foot of solid sand as 161.0 lb. Then 245 lb. of sand has 1.522 cu. ft. absolute volume which gives an *a*/*c* ratio of 3.17. The water per sack of cement is read from Fig. 12, transformed into cubic feet, and added to the volume of cement and sand to give the cubic feet of mortar per sack of cement, the air being zero. By dividing each item in the second column under each mortar by the volume per sack, the volumes per unit volume of mortar are given in the third column. Thus we find $a = 0.530$ for sand D, 0.523 for sand E, and 0.519 for sand C. From these the relative densities are computed using the sieve analyses in Part 1. This is done in Part 2 for sand D.

The value 0.530 is first distributed to

the various size-groups according to the percentage in each as is shown in the first line of Part 2. The second line gives the space available to each group. The largest diameters only can be distributed uniformly throughout the unit volume. Beginning at the right, then, the No. 8 to No. 4 group has available the space left outside the larger particles, or $1.000 - 0.013$, or 0.987. The subspace for No. 14 to No. 8 group is $0.987 - 0.065 = 0.922$, and so on for the smaller diameters. The relative densities are next obtained by dividing the absolute volume of a group by the subspace set down just below it. These values, for d_a, are given in the third line.

The relative densities for the size-groups of sands E and C, computed in the same manner, are shown compared with those of sand D in Part 3 of Table V.

In order to be free from particle interference, d_a should be less than 0.172 (see Table II) so that t will be greater than D_1. The only size-group approaching this concentration is No. 48 to No. 28. This group shows no particle interference for sand D, but considerable for sand E and a greater amount for sand C. This is the order of height of their mortar-voids curves, sand D having the best grading.

Function of Fines in Sands:

Cement paste will leak out of mortars when the void pockets in the sand are too large to hold it. Little geysers bring cement to the surface in flat work, and sand streaks are found in wall surfaces. To correct this condition the void pockets in the sand must be reduced by filling the gap between the cement and sand with finer sand grains to the point at which the largest cement particles can no longer flow out of them freely. The author finds that it is sufficient for this purpose to increase the relative density of either the No. 100 to No. 48 group or the No. 200 to No. 100 group so it is not less than the value given in Table VI.

Applying the criterion of Table VI to the blended sands in Table V, it will be seen that $d_a = 0.046$ for sand D and 0.047 for sand E indicate plenty of fines in the No. 200 to No. 100 group and that $d_a = 0.026$ for sand C indicates too little.

TABLE VI.—MINIMUM VALUES OF d_a TO PREVENT LEAKAGE IN 6-IN. SLUMP MORTARS.

a/c	Round Sand Grains		Angular Sand Grains	
	No. 200 to No. 100 Sieves	No. 100 to No. 48 Sieves	No. 200 to No. 100 Sieves	No. 100 to No. 48 Sieves
2.00	.....	0.075	.....	0.121
2.00	.....	.....	0.026	.....
2.75	.....	0.110	.....	0.170
2.75	0.018	.....	0.035	.....
3.50	.....	0.125	.....	0.194
3.50	0.025	.....	0.050	.....
4.25	.....	0.150	.....	0.230
4.25	0.035	.....	0.062	.....

Adding Fine Aggregate to Mortar:

An ideal blend of two fine aggregates should provide sufficient cohesion to the mortar while permitting the use of the least water-cement ratio for a given slump. Particle interference in sands dominated by one or two sizes is reduced by adding both finer and coarser material. A balance must be effected between too much fines with large boundary effect and an excess of one size-group causing particle interference. Whether any blend is of advantage is best determined from a study of its mortar-voids curve.

In Fig. 2 let the mortar at P to the right of the critical point I be considered.

Let the amount of material to be added increase a/c from k_2 to k_3. If this material is coarse aggregate the point P will move down to 1 on the v/c curve passing through P without it being necessary to add water to the mix, but with sand the boundary effect will cause P to rise above 1 when water is also added to maintain the consistency. If the material added is the original sand, the new mix will be at 3 on the given mortar-voids curve. If the blending sand increases the voids to a point 4 by boundary effect or particle interference, no improvement has been made; a net improvement would give a mortar at some point, 2, below the curve.

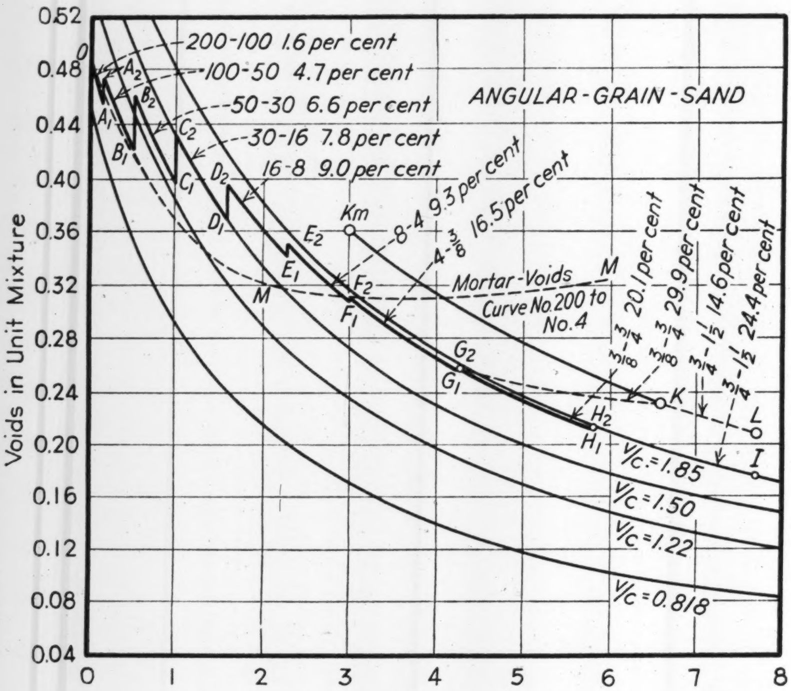

FIG. 13.—Grading the Aggregate.

Adding Coarse Aggregate to Mortar:

In adding particles larger than $\frac{1}{4}$ in. in diameter the increase in surface area is too small for boundary effect to be of moment. Hence if there is no particle interference in the coarse aggregate, the point P of Fig. 2 will move down along its v/c curve without requiring the water-cement ratio to be increased.

Synthetic Composition of the Grading of a Concrete Aggregate:

The principles of grading discussed above are illustrated in Fig. 13. An aggregate assumed to have no particle interference is added to cement paste by successive size-groups beginning with the smallest size, and after each addition water is added to restore the 6-in. slump consistency. The diagram shows the movement of the point of mix from O to A_1 along its v/c curve, then vertically to A_2, the position of the finest sand mortar; with the next size-group the mix moves from A_2 to B_1 and thence to B_2; and so on for the remaining groups. The point I represents the position of the final concrete with $1\frac{1}{2}$-in. gravel.

The mortar-voids curve of the composite sand from No. 200 to No. 4 sieves is given by the curve OMM passing through the point F_2. The boundary effect in the mortar is the sum of the elements A_1A_2, B_1B_2, C_1C_2, etc. For sizes larger than the No. 4 sieve, the boundary effect is not material, so that from F_2 to I the point of mix moves down closely along a constant v/c curve. If particle interference had been introduced into the concrete by increasing the group, $\frac{3}{8}$ to $\frac{3}{4}$ in., at G_2 from 20.1 per cent to 29.9 per cent, this would have caused the mix to rise to a higher v/c curve to the point K, making it necessary to increase the water-cement ratio also. This increase in voids is added to the contained mortar, and if computed by Eq. 2, v_m will plot at the intersection of the v/c curve passing through K with the a/c ordinate passing through F_2, as shown at K_m. The difference in voids between K_m and F_2 measures the degree of particle interference.

CONCLUDING REMARKS

1. The study of the structure of voids in fresh mixtures of cement, sand, and coarse aggregates is important because of the relation of this structure to the water and air contained in the voids.

2. Every change in the grading, consistency, and amount and character of materials causes a corresponding change in the voids in the mortars.

3. All voids in mortars can be traced to the void pockets enclosed by the finest flour particles of cement and to the boundary pockets of these particles at the surfaces of all larger particles.

4. The mortar-voids curve originated by Talbot and Richart offers the best method for studying the changes in voids due to any of the variables affecting mortars.

5. A series of mathematical curves has been added to the mortar-voids chart which serve as reference curves for evaluating and interpreting test data.

6. The chief cause of voids is what is termed boundary effect, which is the difference in percentage of voids in the boundary pockets of the finest particles of a mix at the surfaces of larger particles compared to the percentage of voids in the void pockets of these finest particles. Boundary effect is always present in mixtures of two or more sizes of granular particles.

7. A second and important cause of voids is particle interference between one size-group, present in excessive amount, and particles of the next smaller group size. The critical point at which a size-group with diameter D begins to be in excess depends upon the relation of average clear spacing of the particles t to the next smaller group size, D_1. The author finds that t should be greater than D_1.

8. The mortar-voids chart is employed to distinguish between the voids in mortars due to cement paste, to boundary effect, and to particle interference.

9. In the study of sands by the use of the mortar-voids chart, identical relative consistency of mortars and cement paste is essential in comparing mixtures for each series of data.

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(6) F. O. Anderegg, "Grading Aggregates," *Industrial and Engineering Chemistry*, Vol. 23, September, 1931, p. 1058.

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(10) C. A. G. Weymouth, "Effects of Particle Interference in Mortars and Concretes," *Rock Products*, February 25, 1933, p. 26.

(11) T. C. Powers, "A Discussion of C. A. G. Weymouth's Theory of Particle Interference," Papers of the Portland Cement Assn., Chicago, March, 1936.

DISCUSSION

Mr. F. O. Anderegg[1] (*presented in written form*)—Mr. Weymouth has contributed a thoughtfully constructive paper to the theory of aggregate grading as affecting voids in the concrete. He distinguishes three classes of voids: those in the cement paste, those between the cement paste and the surface of the aggregate due to a "boundary effect," and those due to "particle interference" in the grading, but later the third is combined with the second (conclusion 3). Starting with the cement paste, then any addition of aggregate requires the addition of more water to saturate the absorptive capacity of the aggregate to wet its surface and to fill voids around its periphery due to the boundary effect. The aggregate being a necessary evil, its deleterious effect should be minimized as much as possible by selecting that grading which will reduce to a minimum the boundary effect, to which an excess of any one size, by interfering with the proper behavior of the next smaller size, is probably the worst contributor.

The philosophy on which the writer and Mr. Furnas[2, 3, 5] have developed a theory of grading has been somewhat different. We have proposed to start with aggregate of as large a size as the particular application would permit. The problem then has been to fill in the voids with finer aggregate to as high a degree as possible so as to reduce the amount of binder required. The shape of aggregate particles as well as the boundary effects at the surface of the mortar or concrete product also have been taken into consideration. Two types of grading have been shown to be useful in this connection: a gap grading which permitted maximum packing, but necessitated special means of placing because of harshness; and a continuous grading, which gave pretty good packing and was characterized by excellent workability. This latter property is of great importance in the placing of mortar and concrete and it might well have received more emphasis by Mr. Weymouth. As a portland cement itself is to a very considerable extent aggregate, the grading should be carried down through the fine aggregate and to the finest cement particles. The optimum grading for portland cement seems to be logarithmic.[4] The writer carried out at Mellon Institute in 1928 to 1930 experiments covering these points and since then these principles have been applied in numerous practical problems involving the packing of broken solids.[5, 6] Where special means of placing, such as vibration, are not available, it is important to have a more or less continuous grading so as to provide ball bearings in the right proportion over which the coarser particles can roll. Such a gradation, acting somewhat as does a sand filtration system, also prevents bleeding out of the paste or water. The ratio of sieve fractions, differing in mean dimension by $\frac{1}{2}$, for water-worn aggregate has been shown to be close to 1.2 and for less regular particles to be somewhat less for mortar systems.[3]

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[1] Consulting Specialist on Building Materials, Owens-Corning Fiberglas Corp., Newark, Ohio.

[2] C. C. Furnas, "Grading Aggregates, Mathematical Relations," *Industrial and Engineering Chemistry*, Vol. 23, p. 1052 (1931).

[3] F. O. Anderegg, "Grading Aggregates," *Bulletin*, Am. Ceramic Soc., Vol. 23, p. 1058 (1931).

[4] F. O. Anderegg, "Gestzmassigkeiten in der Kornzusammensetzung des Zements," *Zement*, Vol. 23, p. 99 (1934).

[5] F. O. Anderegg, "Gradings for High Density," *Bulletin*, Am. Ceramic Soc., Vol. 16, p. 11 (1937).

[6] D. S. Hubbell, "A New Inorganic Cement and Adhesive," *Industrial and Engineering Chemistry*, Vol. 29, p. 123 (1937).

The grading proposed more than 30 yr. ago by Fuller approximates fairly well to this value for the sand portion, while for the more recent grading proposed by Bolomy, a ratio of about 1.4 seems to be giving good results. In concrete the total range of sizes from coarse to cement flour is increased several fractions over the mortar range. In portland cement we have about 5 fractions with boundaries at $2\frac{1}{2}$, 5, 10, 20, 40, and at about 80 μ, and here a ratio of about 1.0 seems to give best results. In mortars, the boundaries start at about 80 μ or passing No. 200 sieve, No. 100, 50, 30, 16, also usually No. 8, and No. 4 on occasion (about 5 more fractions) and here the ratio between fractions becomes about 1.2 for best combination of packing and workability. In concretes the coarse aggregate boundaries are usually taken at No. 4, $\frac{3}{8}$, $\frac{3}{4}$, $1\frac{1}{2}$, 3 in., etc., so that a ratio which gives good practical results of 1.4 might seem reasonable.

Another point to be emphasized is that in a great deal of concrete, compromises between gap and continuous grading are often met. Most commercial sands contain an excess of one size, so that the coarse aggregate may be considered as a size unit and the sand, as approximating another size unit, with the cement paste as roughly a third size unit so that something like a 1:2:5 mix should give very good packing.[2] This limit is being more and more closely approached as more effective means of placement are made available; in the most recent work with vibration, the excess of mortar over that actually required to fill the voids in the coarse aggregate has been reduced to about 17 per cent, or about 1:2:4.3.

Lacking such means, however, Weymouth is quite correct in emphasizing the detrimental effect of "particle interference." This has been shown to be specially harmful in mortars,[7] where the element of workability is usually extremely important. Thus in ready-mixed stuccos, the writer has pointed out the importance of having present the proper amount of fine aggregate in the proper grading.[6] But "interference" is regarded by this writer as with the coarser particles rather than with the finer ones. By holding the larger aggregates farther apart than they would lie in a properly graded system, more fines, cement, and water are required to fill the extra voids. The systematic arrangement of different sizes of ball bearings is also disrupted and the workability is hurt. The finer the size "interfering," the more harmful the effect.

Some examples might be cited: a given masonry cement was mixed with an equal weight of water and a commercial masonry sand extracted from the Ohio River was mixed in until a consistency suitable for brick laying was obtained (flow 120). This required only 2.4 times the weight of the cement. With a sand graded so that each coarser fraction should be 1.2 times the next finer one in weight, the amount used was 5.3 times the weight of the cement. In a set of experiments with concrete, all the aggregate was separated and recombined with ratios 1.2, 1.3, 1.4, and 1.5, and it was found that the water requirement to secure a workable consistency increased steadily with the ratio, as did the bleeding tendency of the mixes. In both of these examples, the proper amount of very fine aggregate was carefully added and was proved to be quite helpful.

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[7] F. O. Anderegg, "Portland Cement Finishes," *Journal*, Am. Concrete Inst., Vol. 26, p. 36 (1930).

Weymouth has contributed very helpfully to concrete theory and practice by emphasizing the importance of these fines, and by laying stress on "particle interference" and on "boundary effects."

MR. T. C. POWERS.[8]—Several years ago we started a comprehensive study of mortars to determine whether particle interference had the effect described by Mr. Weymouth. At that time Mr. Weymouth's contention was that when the spacing of a dominant size group became less than the diameter of the next smaller size, there would be a sharp increase in voids in the mix and corresponding strength losses. Furthermore, he believed that strength would decrease out of proportion to the increase in voids. We made a series of tests in which particle interference in sizes from No. 100 up was, as far as possible, the only variable. To eliminate other variables it was necessary to keep the paste content constant, and to do that it was necessary to supplement the cement in the lean mixtures with powdered silica which, at the temperature of the experiment, was considered to be chemically inert.

The results showed that under those conditions in which the texture of the paste itself remained constant, particle interference had virtually no effect; in fact, it had no effect at all at the limit set by Mr. Weymouth. Its effect became evident only when the concentration of one particle size was very much above the limit which he believed to be correct, and then the maximum effect on strength was only about 10 per cent. We concluded that so far as particle interference *per se* was concerned, it was not of very great moment, but we also found that when the cement was not supplemented with silica in the lean mixes, the results were different. There were decreases in strength accompanying increases in particle interference of the order mentioned by Mr. Anderegg, indicating that when the paste itself was of inadequate texture, particle interference in sand sizes had greater effect.

We have under way at present a study in which particle interference and improper texture of the paste are being studied together. We believe that particle interference will appear to be more important under these conditions.

With respect to the boundary effect, we have done no experimental work, but we have some theoretical misgivings. The chart illustrating this theory shows spheres in a state of what is called hexagonal close packing, resting on a plane surface. If, instead of arranging the spheres in this way, they had been shown one on top of the other in a cubic arrangement, the voids at the boundary would have been the same as those inside. It seems, therefore, that if the illustration given is to be applied to concrete, the particles forming the boundary voids must approach the condition of hexagonal close packing. Mr. Weymouth says that the boundary voids in a concrete mix are formed entirely by the smallest particles, for if there were boundary voids formed by larger particles, the smaller particles would enter and fill these voids, forming new and smaller boundary pockets. A computation of the spacing of the finest particles based on the volume of water usually present in concrete will show that the average spacing of those particles is such that they could be arranged in the cubic arrangement without changing the volume of voids. It would seem, therefore, that there could be no boundary effect at water ratios encountered in concrete.

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[8] Assistant to Director of Research, Portland Cement Assn., Chicago, Ill.

MR. F. E. RICHART.[9]—I believe this paper is an excellent piece of work and that it gives new information on the arrangement of the particles in a mortar or concrete mixture. Several years ago I made some studies that are pertinent to this discussion. Most of the information is given in the accompanying Fig. 1, in the form of rational lower limiting curves or envelopes which definitely fix the minimum possible values of the mortar voids made with a given sand. These curves have been drawn for the mortars of maximum density, or basic water content, which correspond to a rather dry mix, but similar curves could be drawn for wetter consistencies.

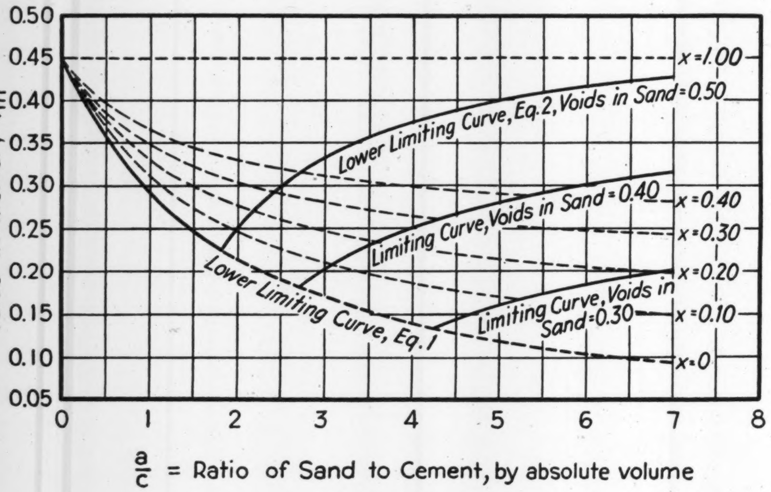

FIG. 1.—Mortar-Voids Curve.

Refer first to the left-hand group of curves. The minimum voids in a neat cement mortar are about 0.45. If sand particles are added to the paste, the percentage of voids is obviously reduced as indicated by the lower curve. If v_m denotes the proportion of mortar voids, v_n the value of the voids in neat cement paste, and a and c the absolute volumes of sand and cement, respectively, then for a mortar in which there is sufficient paste to completely surround the sand particles, the value of v_m is as follows:

$$v_m = \frac{v_n}{1 + \frac{a}{c}(1 - v_n)} \quad \ldots\ldots(1)$$

For the case in which the sand particles are incompletely surrounded by cement paste, consider first the sand alone as a mortar with an infinite value of $\frac{a}{c}$. Such a sand, with enough moisture to produce minimum bulking (basic water content) will have voids denoted by v_a. Now if cement particles are added to the sand in such a way as to fill up the sand voids, the value of v_m will comprise the voids in the sand minus the absolute volume of the cement particles. For this condition,

$$v_m = v_a\left(1 + \frac{c}{a}\right) - \frac{c}{a} \ldots\ldots(2)$$

Equation 2 applies to those mortars in which there is not sufficient paste to surround the sand particles. The intersection of curves representing Eqs. 1 and 2 in Fig. 1 represents the mixture in which the cement paste just fills the sand voids, for which the value of

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[9] Research Professor of Engineering Materials, University of Illinois, Urbana, Ill.

$$\frac{a}{c} = \frac{1 - v_a}{v_a(1 - v_n)}$$

Equation 1 presupposes that the sand particles are large, so that the cement paste will envelop them without an appreciable "boundary effect." If the sand particles were as fine as the cement, the voids in the sand mortar would be nearly as great as in the cement paste. Hence Eq. 1 will not apply if the sand contains very fine particles itself. One might consider that the material passing the No. 100 sieve, while not as fine as cement, would produce nearly as great voids as the cement. Treating this very fine sand as if it were cement, a group of curves similar to that of Eq. 1 are found. For these curves, x represents the ratio of 0-No. 100 sand to the total sand sample. As x increases, the position of the limiting voids curve is raised in Fig. 1.

A comparison of these analytical curves with actual mortar-voids curves shows fairly good agreement, the test curve, of course, always giving higher values of the mortar voids. The difference between the two is due naturally to boundary effects or "particle interference." With the wetter consistencies or with mechanical placing aids such as vibration, the agreement is better than for the very dry mixes.

Three typical mortar-voids curves for sands may be visualized from Fig. 1. (*a*) A very fine sand will have rather high mortar voids for all mixes, since x is large and v_a is large. (*b*) A coarse, one-size sand, like Ottawa standard sand will have fairly low voids in rich mortars but rather high voids in the leaner mixtures. (*c*) A well-graded torpedo sand, having low values of both x and v_a, will exhibit relatively low values of mortar-voids over a wide range of ordinary mixtures. It is obvious that much can be foretold regarding the concrete-making properties of a sand from the sieve analysis alone, though the amount of divergence from the analytical curves requires tests or further studies of the sort that Mr. Weymouth indicates.

MR. ROY W. CARLSON.[10]—It may be well to give an indication as to how great the effect of particle interference may be in ordinary concrete. In my paper on drying shrinkage,[11] concrete mixes were tested in which the variable was particle interference, which was introduced by having an excess of a single sieve size of aggregate. In these tests, concrete having an ideal gradation, in which there was a systematic variation in the percentages of successive sizes of aggregate, was compared with three concretes in which there was about three times the ideal amount of a single size of aggregate. The result was that the concretes having particle interference required from 3 to 6 per cent more mixing water than did the concrete having an ideal gradation. The early shrinkage was greater and the early strength (not shown) was less for the concretes having particle interference but the differences were only about as much as would be expected from the increase in water-cement ratio. Long-time strength and shrinkage were affected only slightly, despite the greater water-cement ratio. One must conclude that as far as strength and drying shrinkage of ordinary concrete are concerned, particle interference is not as big a factor as has been indicated from tests on mortars.

MR. W. F. KELLERMANN.[12]—As I

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[10] Associate Professor of Civil Engineering, Massachusetts Institute of Technology, Cambridge, Mass.

[11] Roy W. Carlson, "Drying Shrinkage of Concrete as Affected by Many Factors," see p. 419.

[12] Associate Materials Engineer, U. S. Bureau of Public Roads, Washington, D. C.

understand Mr. Weymouth's method, after the proportions are worked out for a given sand, it is possible to interchange coarse aggregates without materially changing the mix provided the coarse aggregates have approximately the same grading and percentage of voids. We do know, however, that when designing for flexural strength the mineral make-up of the aggregate may affect the design by half a sack of cement per cubic yard. Therefore I should like to ask Mr. Powers whether he thinks that we can disregard the mineral make-up of the coarse aggregate.

MR. POWERS.—Personally, I do not think so and have not gained that impression from Mr. Weymouth's work. Mr. Weymouth's method is, in my estimation, not primarily one of design; it is one of diagnosis. By means of the computations given, he can locate the faulty part of a mix and prescribe a remedy. That is a relatively new departure in the field of mix design—diagnosis and prescription, rather than prediction.

It should be noted that Mr. Weymouth's work does not deal with the relation between water ratio or void ratio and strength; it deals with factors of mix composition which control the water or void ratio. What the strength at a given water ratio may be depends on many other factors.

MR. THADDEUS MERRIMAN.[13]—If a moral were to be drawn from this paper, it would be that no mistake can be made by always using plenty of cement and, as a corollary, it goes without saying that the better the cement, the better the resulting concrete will be. There is so much variation in quality between the output of various mills that the differences due to this cause far overshadow all matters of relationship between voids and surface boundaries, of percentage of water used, of the ratio between the cement and the sand and many other similar and obvious fundamentals. The ingredient which makes concrete is the cement. Without cement there would be no concrete. In order to make better concrete we must have better cement of greater uniformity.

Of first importance also we must learn why one cement makes concrete that is hard, dense, rock-like and durable, while another produces soft, chalky concrete which disintegrates readily. The cause of concrete will not be advanced until these basic relationships have been established. Little is to be gained by working abstractly with cements whose characteristics are unknown.

MR. H. J. GILKEY[14] (*by letter*).—Cases of pronounced particle interference in concrete are familiar to all of us in the raw, harsh, bleeding type of mixture that fortunately has been largely replaced by the fatter, somewhat oversanded concretes currently used.

One practical contribution of this paper is that it calls attention to the importance of suitably graded fine aggregate and to the fact that some of the ills of concrete, notably that of bleeding, can be avoided with proper attention to the grading. Many of the engineers and technicians are only beginning to realize that not all fines are "dirt" and that in the majority of cases current concrete sands have been over-washed.

The technique of grading, which the author here expounds, promises to be an outstanding contribution that will carry this important phase of concrete a long step forward. For collateral reading, the writer strongly recommends pages 86–93 of *Bulletin 137* (**2**),[15] for the general background which it supplies. He also urges that anyone not familiar with Weymouth's earlier work secure and study the very lucid discussion by T. C. Powers, listed by the author as reference No. **11**. Without the groundwork of Powers' discussion (**11**), the writer fears that many will find this paper difficult.

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[13] Consulting Engineer, Board of Water Supply, City of New York, New York City.

[14] Professor and Head of Theoretical and Applied Mechanics, Iowa State College, Ames, Iowa.

[15] The boldface numbers from 1 to 11 in parentheses refer to the reports and papers appearing in a list of references appended to the author's paper, see p. 372; the references numbered from 12 up are appended to this discussion, see p. 393.

While a primary aim in mixing concrete is to produce a material which will develop strength, no actual strength data or comparisons are included within the scope of this paper, the implication properly being that better grading will produce better concrete, and that better concrete will be so whether comparisons be on the basis of strength, economy of cement, workability, durability, or what not. Within the range of comparable mixtures such an assumption is valid. Moreover, any improvement in grading is all to the good.

The primary point which the writer wishes to make in this discussion is that, in spite of its many possible benefits, grading is not a panacea that can iron out differences between non-comparable mixtures. Powers (**11**, p. 9) expressed a similar skepticism.

The grading, however perfect, cannot eliminate water gain (although it can greatly diminish it), nor can it offset inherent differences between a concrete and its mortar that make quite untenable the venerable concept that the strength of a concrete is the strength of its mortar. While the two have much in common they are two different types of mixture, as are different concretes composed of different quantities and size ranges of aggregate. Until these distinctions are frankly recognized and included in the reckoning, we shall continue to design mixtures on a false premise and fail correctly to understand concrete and its behavior under load.

One of the most encouraging statements the writer has seen is that by Mr. Powers (**11**, p. 24): "Regardless of the merit of the theories propounded in the foregoing, there is established one undeniable fact, *the grading of the fine aggregate can have a very large effect on strength*, and probably other properties too. The indications are that *at a constant water ratio in the range of those of ordinary concretes, as much as one-half* of the theoretically possible mortar strength can be lost through defects in grading alone."[16] (The italics are the writer's.)

For one in Mr. Powers' conservative environment to recognize and to concede that within the realm of what we have always accepted as workable mixtures, there can be really important variations of strength within the "water-cement ratio law," seems at last to indicate progress toward freeing concrete from the unreasoning domination of an over-statement made sacred through repetition and wide dissemination. If "within the law" the fine aggregate can effect strength, the coarse aggregate can conceivably do likewise, which fact may yet raise to the level of respectability an ever-increasing mass of evidence supporting the contention that *the strength of a workable concrete is not usually, and probably never is, the strength of its mortar.*

To understand why certain inherent differences between a concrete and its mortar have so long gone unrecognized and their possible influences unquestioned, necessitates a brief résumé of the current philosophy of mixture design with special reference to the background of its development.

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[16] Powers is not alone in his recognition of the high strength fluctuations which are possible "within the law." A recent bulletin by Thomas and Graf (**21**, Table 4) shows a range of compressive strength of from 59 to 131 per cent of the average for mortars from 30 different Oregon sands at constant water-cement ratio and equal flow. The same bulletin, Table 5, Part B (**21**, p. 39) shows that the presence or absence of silt in the sand may produce great strength fluctuations at constant water-cement and sand-cement ratios. The great differences in strength attainable through differences in grading alone are also strikingly shown in data reported by Committee C-1 (see reference **22** and discussions). But differences in grading are not the only cause of variations "within the law" (**23**, Table 2 and Fig. 5) for here a single aggregate at a constant c/w ratio of 2 (Mortars A and C) and identical cements, gave great strength fluctuations at ages of 1, 3, 7 and 28 days. The only difference between the A and C mortars was in the amount of aggregate which was necessary to secure the constant flow for mixtures C in comparison with the fixed aggregate-cement ratio of Mortar A (2.75). In limited reconnaissance tests a few years ago the writer found that by adding granite crusher dust to an otherwise constant mixture, it was possible to introduce rather large strength differences "within the law."

1. Acceptable concrete consists of a cement matrix greatly expanded by the insertion of firm, hard, inert particles (aggregate) at least as strong and as durable as the matrix; the aggregation, as mixed, to be plastic and placeable. Basically, the matrix is, of course, the cement and water paste, but some think of the matrix as being the sand-cement mortar to which coarse aggregate is added as a diluent. The difference is immaterial to the present discussion.

2. The strength (and the weakness) of the mortar, or the concrete, is the strength (and the weakness) of its paste.

3. The inherent quality of the paste is inversely proportional to the dispersion of its cement particles. The spaces between particles are filled initially with water and a little entrapped air, summarized as voids.

4. For a paste of given quality the most economical concrete is that which contains the least paste (and the most aggregate), and here is where the matter of grading has up to the present time entered the picture. The economical aspect of grading has been well illustrated by the story of the full barrel of apples between which could be packed nuts, between which could be packed respectively peas, rice, and flour.[17]

The foregoing statements are representative of concrete orthodoxy up to 1938. If they are all correct, it follows that the quality of *workable* concrete is dependent solely upon the quality of the paste and within the range of *workable mixtures* is independent of the size, grading, and amount of aggregate used with a given paste.

So much for *quality*. Economy is a different matter and one in which grading, especially of the fine aggregate, plays an important part. For the water-cement ratio criterion, that aggregate is most economical (and therefore the best) which requires the least ratio of water to cement per unit volume of concrete. In like manner for the voids-cement ratio criterion, the most economical aggregate will show the least ratio of voids to cement as determined by trial. Neither theory concedes that the number, shape, and grading of the aggregate particles are important except as they influence the water requirement or the voids. Aggregate is simply inert filler.

Weymouth (and Powers) (**10**) (**11**, pp. 12, 16, 24) have advanced their theory to include the grading as being significant in its own right, and not merely as an adjunct to economy. They have pointed out the importance of the fines in subdividing the water and trapping it in compartments small enough to retain most of it. The same thing that prevents bleeding greatly reduces[18] the water-gain (**12**) and thereby improves the uniformity, strength, and general quality. This represents a distinct advance. While further recognizing that the importance of the grading is not limited to the mortar, Weymouth has failed to recognize important inherent differences between concretes and their mortars. In this respect he tends to perpetuate a fallacy of long standing.

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[17] By increasing the maximum size of aggregate and improving upon the excellence of the grading the potentialities of a sack of cement become such as to fire the imagination of a Scotchman. Experience has demonstrated, however, that as the paste deposits become smaller (more aggregate to be surrounded and smaller passageways between particles) greater fluidity of paste is required to supply the mobility, to the mixture, which is necessary for placement. Thus the ratio of water to cement must be increased for the leaner mixtures and the quality (strength) of the paste, and its concrete is lowered thereby. This is well illustrated by the author's Table I, which shows successive water-cement ratios of 0.54, 0.59, 0.69, and 0.81, for mortars all with a 6-in. slump but of proportions ranging from 1:1 to 1:2½. Of course these increases are over and above those necessary to satisfy absorption of water by the additional sand. The stiffening influence of coarse aggregate on the plastic mixture is exactly the same in kind but increasingly less in degree as the size increases; for in this regard fine aggregate is "simply coarse aggregate grown small," in spite of the contrary implication on p. 358. Nevertheless the better the grading the denser will be the concrete and the greater will be the yield secured from paste of a given quality.

[18] Water gain can be greatly reduced but it cannot be eliminated in any workable mixture properly placed. Adequate placement requires that capillary continuity be established throughout the mass, as is indicated by the sheen of moisture that gathers at all top surfaces of properly placed fresh concrete. That this moisture layer (and air bubbles too) on the underside of aggregate particles (large and small) interferes with the bond, is evidenced by the adhesion of the aggregate particles to all top surfaces as cast, whenever hardened concrete is fractured along planes which were horizontal as cast (**12**).

As noted previously, the writer cannot visualize perfection of grading as being able to eliminate internal stratification or water gain in anything like its entirety. Moreover, concrete will still be concrete with the inherent weaknesses of any material composed of coarse particles which introduce abrupt discontinuities. These, either under load or under volume changes, introduce stress concentrations and bonding problems. As the sizes of particles increase these differences are magnified.

Let us here refer again to the writer's statement No. 2, under the philosophy of current mixture design: "The strength (and the weakness) of the mortar, or the concrete, is the strength (and the weakness) of its paste."

It is not surprising, perhaps, that the earliest of our theorists should have convinced himself that the strength of an aggregation should be at least that of its weakest constituent. It is amazing, however, that once this postulate was laid down, practically no one, among all the experimenters in the great field of concrete, should have had enough curiosity or skepticism seriously to have attempted to check its validity experimentally.[19] The orthodox view (1938 version) continues to be that of statement No. 2. The writer maintains that in the light of current knowledge of how materials behave under load, the statement is illogical.

Examine, for example, a block of concrete. It is a structure made up of two types of members: particles of aggregate and accumulations of hardened paste. When the block of concrete fails, the primary yielding or incipient failure must occur:

(*a*) In a particle of the aggregate,

(*b*) In the paste, or

(*c*) At the junction of paste and aggregate.

Statement No. 2 takes no cognizance of the fact that the bond between the aggregate and the paste may be the "Achilles heel" of the combination.

The steel framework of a building consists of girders and columns, but its strength may lack much of being the strength, either of the girders or of the columns.

Had our original thinker thought somewhat less realistically and visualized his concrete as rubble masonry, set up with a portland cement mortar, he might have had some qualms.

Or had he visualized his diluting material as a lot of polished marbles with greasy surfaces, he might have uttered statement No. 2 with less assurance, and others might have built more cautiously thereon.

During the fifteen years since the attention of the writer was first attracted to the adverse effect of water-gain (12) on the bond beneath particles of aggregate, there has come an almost universal recognition of the phenomenon both as regards aggregate and reinforcing steel.

But how slowly do our questioning processes evolve! At last we recognize and concede that the bond on bottom surfaces is less perfect than on top surfaces, but how many persons have, because of this, begun to have misgivings as to whether or not the strength of the concrete is the strength of its paste?

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[19] There have been checks, of course, many of them, but the writer, from his biased point of view as "one of the heretics" has felt that some of the experimental checks conducted by the "orthodoxy" have been "lawyers' checks" designed not so much to open up the question as to keep it closed.

And has anyone at any time audibly raised the question, "How much bond is enough to insure that the strength of the concrete is the strength of its paste?" And is the bond between paste and aggregate anything like the same problem for particles of small size that it is for large stones?

There are, of course, many respects in which the strains and stresses to which a block of concrete is subjected differ from those to which a similar block of hardened paste or a block of mortar would be subjected under exactly the same external environment. These are due to such inherent internal conditions as amount of heat generated, volume of water taken on in curing, or volume lost in drying out. These things cannot be gone into here, but they, too, are a part of the picture, although generally recognized as being relatively much less important in comparisons of small test specimens than they would be in comparing full sized structural members.

The point to be emphasized here is that, even if ideally graded, there would still exist between a concrete and its mortar (and even more its paste) fundamental differences of a kind that would have to be averaged out by a remarkable balancing of opposing factors to permit the two materials even approximately to equal one another in strength, durability, or other essential respects.

From a great many comparative tests of concretes and their mortars[20] the writer has yet to find a mortar that was not from 15 to 50 per cent stronger than its concrete under similar conditions of placement, curing, and testing.[21] Representative results from these tests and more detailed discussions of many of the points involved in such comparisons can be found in some of the papers appearing in the attached list of references (**13, 14, 15, 16, 17, 18, 19**). The discussions under references 13 and 14 bring into the open an unusually large number of the aspects which tend to befog test data and give rise to valid differences of opinion as to what a given set of results really shows.

In reference 18 is found an elaborate and admirable series of tests purporting to investigate the effect of size of aggregate upon strength, but unfortunately the three variables simultaneously present (size of aggregate, size of specimen and quantity of aggregate) were so intermingled that violent interpretational differences have arisen which leave the authors of one belief, the discusser of an opposite belief, and the rest of the interested concrete constituency compelled to dig out their individual beliefs or to take, on faith, the very positive utterances of one or the other of the parties to the controversy.

All of the foregoing evidence is on differences observed in ultimate compressive strengths. Only recently the writer and Glenn Murphy, of this department, have extended their studies to comparisons of the relative stress-strain behaviors of mortars and concretes during test, and they are discovering some most consistent and interesting evidence that the progressive breakdown process, as loads are increased, is basically influenced by the quantity and the grading of the aggregates more than by others of the variables investigated. Evidently the observed differences in ultimate strength are not at all due to something that happens just as the ultimate load is reached but are the result of a progressive breakdown that shows first on the stress-strain curve at not over 50 per cent of the ultimate load carried. The symptoms are such as to lend credence to the theory that specimens fail initially by slippage between mortar and the larger surfaces of the aggregate particles.

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[20] By the mortar of a concrete is meant the product, much more fluid than the concrete, that results if the coarse aggregate is either removed or omitted, and not such a product thickened up by adding more fine aggregate as has sometimes been done in alleged comparisons of the two (**17**, pp. 405 and 410). Adding fine aggregate may alter the strength even more than would adding an equal weight of coarse aggregate. Under this condition one is simply comparing the concrete with a lean mortar.

[21] In practically all of the writer's published comparisons, the equal water-cement ratios of mortars and concretes are nominal ones to which the aggregate of the concrete was added dry. There is always an appreciable absorption of water by the dry, coarse aggregate, which lowers the true water-cement ratio of the concrete relative to that of the mortar. The true difference in favor of the mortar is invariably from 5 to 20 per cent greater than that indicated by the nominal comparisons. The comparisons were purposely made on this basis in order to demonstrate the substantial difference even when absorption favored the concrete.

The paper presenting these results (20) is commended for study by such persons as may be interested in determining for themselves whether or not statement No. 2 is a valid one, and whether or not any system of grading can be expected to result in making concretes behave like and develop the strengths of their mortars.

In closing, the writer wishes again to commend the author upon the fundamental nature of this promising venture. He wishes also to emphasize that the inherent differences between concretes and their mortars, while academic, no doubt, so far as the practitioner is concerned (for no sane person is going to substitute mortar for concrete) are real, nevertheless, and must be recognized and analyzed if concrete is to be understood. Eventually, extension (and correction, if necessary) of the theory will benefit the art and improve the practice. Nothing is to be gained by attempting to perpetuate a fallacy, however venerable it may have become.

The writer hopes that the author will continue his grading studies to determine more fully, on a groundwork of demonstrated fact, what may be the ultimate benefits which can be secured from improved gradings of acceptable aggregates. The study will also bring out whatever there may be in the nature of inherent grading limitations. In conducting the study it is immaterial whether the author believes initially that the potential strength of the concrete is, or is not, the strength of its mortar, since that question will be answered eventually on the basis of experimentally determined fact.

MR. DUFF A. ABRAMS[22] (*by letter*).—Mr. Weymouth has introduced a number of new concepts. The writer wishes to examine these in the light of what we already know of the properties of concrete aggregates and the resulting concrete. Are these new concepts conducive to a better understanding of concrete mixtures? How do they fit into the concrete *credo* as established by earlier studies?

In any discussion of concrete mixtures it is important to bear in mind that the "mortar" portion is not an independent entity, but is simply an arbitrary division of the aggregates at a certain point; this point is largely a matter of latitude and longitude. In France the division between fine and coarse aggregates is made on the 5-mm. round screen; in Germany a 7-mm. round opening is used. In this country we generally make the separation on the No. 4 square-mesh sieve (0.187 in. or 4.70 mm.); this gives a size a little larger than French practice.

The writer's investigations 20 yr. ago were probably among the first to bring out the fact that sands of widely different size or grading can be used with satisfactory results, if each is mixed in concrete to its optimum percentage. The practical result is that the finer sands must be used in lower percentages than the coarser sands. The same studies brought out the fundamental relations of the water-cement ratio to concrete strength and other properties.

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[22] Consulting Engineer, New York City.

Many serious mistakes have been made and much confusion has resulted from attempting to interpret the evidence of sand mortar tests in terms of concrete. The same sand may be prepared of widely different sizes or gradings which will exert an important influence on the properties of mortars made from such sands; however another factor enters into the concrete, namely, the *percentage of sand used.* By adjusting this percentage to secure the optimum grading of the combined aggregate it is feasible to make equally good concrete from any of a wide variety of sizes and gradings of sands.

The void-cement ratio attempts to take into account also the small quantity of entrained air; however it should be pointed out that the entrained air in mortars is not a measure of entrained air in concrete.

Let us refer to some of the features of the paper.

In the author's original paper[23] it is stated that particle interference explains the deviations of concrete strength from the water-cement-ratio law; in the present paper the same function is used to explain the higher water requirements of concrete of unsatisfactory aggregate grading. It is difficult to reconcile these two conflicting views.

The author does not state in so many words, but we may imply that his object is to reduce voids in the concrete to a minimum; in other words to produce a concrete of maximum density. I use the word density here in its commonly accepted meaning as referring to the total solids in a unit volume of concrete.

There will probably be no objection to using strength and permeability of concrete as satisfactory criteria of its suitability. What are the facts with reference to density? Many investigations have shown that we must be on our guard in any off-hand conclusions with reference to the relation between concrete strength and density; in fact the densest concrete is generally a very lean mix, that would not usually be satisfactory from the standpoint of either strength or permeability. The case is illustrated by the following values based on tests (quoted by McMillan in "Basic Principles of Concrete Making," p. 44, McGraw-Hill Book Co., New York City) of concrete of the same sand and gravel and the same workability as measured by the flow test:

Mix by Volume	Voids, per cent	Water-Cement Ratio, w/c, gal.	Approximate 28-day Strength
1:3	15	4	6000
1:5	15	5	4000
1:7	15	8	2000

This shows that the cement paste is the least dense portion of the concrete. Permeability tests are not available on these mixtures, but it seems probable that permeability would be governed largely by the water-cement ratio.

Suppose we use the same cement content and vary the density by changes in the size and grading of aggregates and the necessary changes in water-cement ratio. So long as we use workable mixes and unless we go too far afield in aggregate grading we find that strength is a function of water-cement ratio and that this function is a sufficiently accurate measure of density; at any rate the density varies over a narrow range and it is seldom practicable to work to the maximum density.

What about permeability in the latter case? Tests by the U. S. Bureau of Reclamation have shown that density is a false guide as to permeability of concrete, as illustrated by the following values, interpolated for a constant water-cement ratio of 0.66 by weight; slump about 4 in.:

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[23] C. A. G. Weymouth, "Effect of Particle Interference in Mortars and Concretes," *Rock Products*, February 25, 1933.

MIX	APPROXIMATE VOIDS, PER CENT	PERMEABILITY COEFFICIENT
Neat cement	70	300
0–No. 4 sand mortar	29	70
0–¾ in. concrete	18	200
0–1½ in. concrete	16½	390
0–3 in. concrete	16½	650
0–4½ in. concrete	13½	950

The foregoing values are from Fig. 14, *Journal*, American Concrete Institute, January–February, 1936, p. 385.

The tests cited lead us to wonder just what value Weymouth attaches to securing a concrete of maximum density, assuming that it could be secured without undue cost.

Weymouth introduces the concept of boundary effect in a manner that is far from convincing and which does not appear to agree with observed facts. If we mold a mass of neat cement, mortar or concrete against a smooth surface, say plate glass, we fail to find anything which has the remotest resemblance to the condition illustrated in his Fig. 3. It may be claimed that this is a submicroscopic phenomenon which is not visible to the eye; but it appears to the writer that our concepts of viscosity and internal friction are sufficient to account for the fact that additional water is necessary when we add more sand to a mortar mix. It is exactly the same phenomenon that we observe when we note that a viscous liquid flows more slowly through a small orifice than it does through a large one.

Weymouth's concept of particle interference would be more convincing if he had not included the case of the mixture of 2 sizes of dry gravel (Fig. 5). Even in the case of gap grading (No. 4–⅜ in. and ¾in.–1½ in.) there is no sharply-defined point of maximum density. We have heretofore applied the term "random packing" to this condition; this term seems adequate and is well understood.

The relationship of random packing of sand and gravel of various sizes has been fairly defined by the reports of Walker and others. A mixture of sand, say, 0–No. 4 and gravel, No. 4–1½ in., does not show any well-defined point of maximum density; in fact over a range of sand from about 35 to 60 per cent, there is little or no change in the density of the mixture.[24] If this is a fair example of particle interference, it would not seem necessary to compute particle distances to one tenth of a millimeter.

We know that in a concrete mix the aggregate particles of a given size range are seldom or never uniformly distributed. How does it happen that the plastic concrete can be so sensitive as to indicate at once when particle interference occurs as visioned by Weymouth? There are openings between aggregate particles (of a given size range) much larger than those defined by their average distance apart; since we are dealing with a random mixture of particles which do not normally touch each other at all, it would appear that the influence of particle interference would be less sharply defined here than was the case of the dry sand and gravel mixtures mentioned above.

The writer has examined dozens of sawed sections of concrete and scores of photographs of such sections and cannot recall a single instance in which 2 pieces of coarse aggregate were in contact.

Much concrete which shows particle interference as visioned by Weymouth gives entirely normal and satisfactory results in practice and in tests. The case of over-lap gradings, (for example sand 0–⅜ in. mixed with gravel No.

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[24] Stanton Walker, "Effect of Grading of Gravel and Sand on Voids and Weights," *Circular 8*, National Sand and Gravel Assn. (1930).

4–1½ in.) would seem to present a flagrant case of particle interference, yet mixes of this type have been found to behave entirely normally as indicated by both workability and strength.

The case for particle interference seems to lack much when viewed in the light of our previous knowledge of concrete.

Just a few more observations as to certain details of the paper. Sieve analyses are frequently reported to $\frac{1}{10}$ of one per cent. Needless to say no such refinement is justified and is entirely ridiculous in view of the facts as they exist. Tests carried out by A.S.T.M. Committee on C-9 on Concrete and Concrete Aggregates on 100 grab samples of sand from the same cargo showed that the "coefficient of variation" on the standard screens ranged from 15 to 42 per cent; the coefficient of variation for the fineness modulus of the 100 samples was 5.36 per cent. The subcommittee report[25] states:

"If the laboratory technicians are willing to accept the fineness modulus as an adequate factor for expressing the size complex, these data show that in cases where the standard deviation divided by the mean of any series of tests does not exceed 5 per cent, the results are as accurate as would be expected from 100 analyses."[25]

Even where sands are separated into 3 size components and recombined in an effort to maintain a uniform grading, it is not uncommon to find variations up to 3 per cent on certain sieves.

Tests quoted by Dunagan[26] showed that about 50 samples of fresh concrete must be analyzed in order to get within 3 per cent of the known quantity of sand in a concrete mix.

The writer does not mean to imply that such variations are injurious to the concrete, but wishes to emphasize the absurdity of attempting to deal with aggregate gradings to any such refinement as that used by Mr. Weymouth.

This paper seems to go too much to ratios; many of the factors could have been indicated more directly and much more clearly by dealing with actual quantities of cement and water or cement and sand. Figure 10 is an interesting case and shows how tricky ratios may become. The author apparently failed to note that v/c and w/c are not independent variables. In fact if we disregard small percentages of air, v and w are the same thing; the diagram then becomes simply a 1 to 1 relationship between water as ordinates and water as abscissae.

It is regretted that the author did not give more data on the make-up and properties of the 2 concrete mixes in Fig. 13. What was the nature of the coarse aggregate? Was the sand below No. 200 screened out? What was the slump and strength of the 2 mixes?

In the writer's method of design of concrete mixes proposed 20 yr. ago,[27] the upper limits of the "coarseness" of the mixed aggregate was defined by the quantity of cement and certain upper limits for the fineness modulus which was determined by tests. The best estimate I can make of the mixes in Weymouth's Fig. 13 leads me to the conclusion that they are both too coarse for the quantity of cement used; this was the 1918 model of what Weymouth terms particle interference.

The paper seems to be of extremely limited application in that it is based on a uniform slump of 6 in. A large proportion of concrete today is being placed by means of internal vibration, in which concrete of slumps of 1 to 2 in. is satisfactorily placed. In fact much mass concrete is mixed to a water-cement ratio below that which shows any slump. Here workability has ceased to exist except for the few seconds the vibrators are effective. What is the interpretation of particle interference in mixes of this kind?

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[25] Report of Subcommittee XI, Committee C-9, on Evaluation of Data, *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 271 (1935).

[26] W. M. Dunagan, "A Proposed System for the Analysis and Field Control of Fresh Concrete," *Bulletin 113*, Engineering Experiment Station, Iowa State College (1933).

[27] Duff A. Abrams, "Design of Concrete Mixes," *Bulletin 1*, Structural Materials Research Laboratory, Lewis Institute, Chicago (1918).

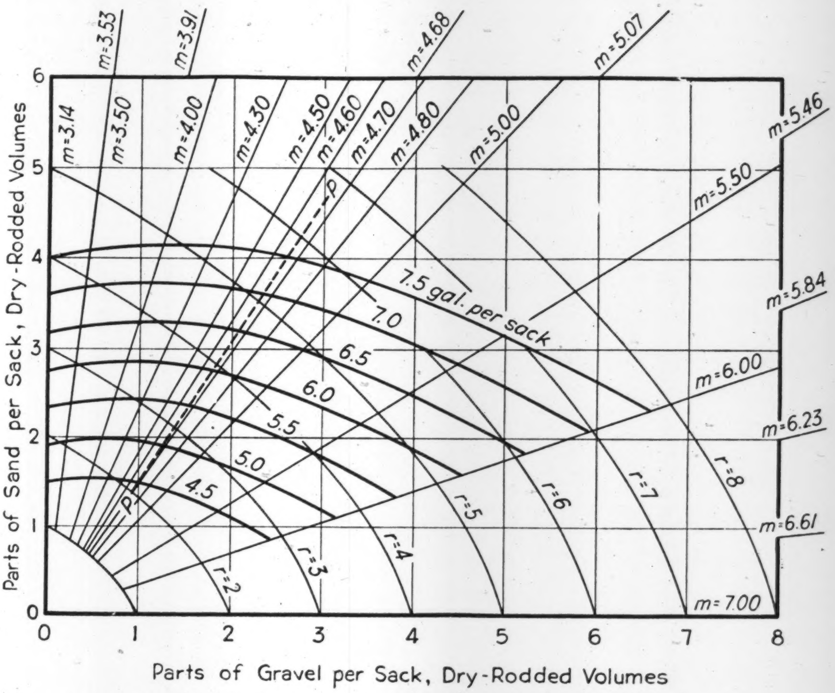

FIG. 2.—Water-Cement-Ratio Relation by Duff A. Abrams' Formula:

$$x = 1.1\left[\frac{3}{2}p + \frac{0.30\,r}{1.26^m}\right].$$

m = Fineness modulus. r = "Real mix" (ratio of volume of mixed aggregate to volume of cement).
p = Normal consistency of cement, ratio by weight.

MR. C. A. G. WEYMOUTH[28] (*author's closure by letter*).—Mr. Abrams opens his criticism by saying that the mortar portion of concrete is not independent but is simply an arbitrary division of the aggregate at a certain point, or grain size. The implication is that mortar needs no special consideration. If that were true there would have been no reason for writing the paper under discussion. However, it is the author's firm conviction, resulting from the analysis of thousands of tests, that it is not true. It is his view that concrete is mortar containing coarse aggregate and that the characteristics of the concrete are largely determined by the characteristics of the mortar.

In support of this, the accompanying Fig. 2 has been prepared by plotting Abrams' (**5**)[29] formula for the water-cement ratio, omitting the water of absorption items, taking parts of sand and of coarse aggregate as coordinates (to be combined with one part of cement for a relative consistency of 1.1). The sand and gravel are those of series 186, of the Portland Cement Association. The water-cement ratio lines are the curves so marked, which are nearly horizontal, and almost parallel the mortar lines until they reach the line *P-P*, when they turn downward. This change in direction shows a critical condition in the mixtures.

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[29] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to the paper, see p. 372.

The parallelism of the mortar lines and the water-cement-ratio lines shows that the addition of coarse aggregate causes no change in the consistency until the point on *P-P* is reached. Up to this point the mortar governs the consistency. Beyond the line *P-P* the downward trend shows that more water must be added to retain the consistency, increasing the water-cement ratio.

The line *P-P* marks where the addition of coarse aggregate produces particle interference. The author confirmed this in the laboratory by mixing coarse aggregate gradually into mortar to ascertain the proportions causing an increase in water-cement ratio and then studying the average spacing of particles at this point in the dominant coarse-size group. He found for all variable gradings of coarse aggregate tried that at this point the average distance between these particles is equal to the average diameter of the particles in the next lower size group, or where $t = D_1$.

The line *P-P* also offers a criterion of workability: the additional mixing water required by those mixes which fall below it make wet looking concrete in which cohesion is greatly reduced so that coarse aggregate separates readily from the mortar. The increase in economy *for a given cement mortar* may be obtained without increase of water-cement ratio by blending available coarse aggregates so as to increase the coarse size groups to just below particle interference, judged by Table II. This has the effect of moving the line *P-P* to the right.

Abrams assumes that the author designs concrete for maximum density. In discussing the blending of sands, the statement is made that an improvement in mortar results in lower mortar voids, inferring a lower water-cement ratio. But this implies not a greater over-all density but an increase in the density of the cement paste. Whether over-all maximum density, using a given mortar, is desirable depends upon placing conditions, and whether the cement saving resulting from maximum density is true economy depends upon the workability needed, the relation of maximum particle size to the spacing of bars, and other considerations.

In considering Fig. 5, Abrams apparently misunderstands the function of the upper curves which he discusses. Their purpose is merely to provide data for fixing points upon the lower curves. As described in the paper, it is only the lower curves that show where particle interference begins by diverging from the straight lines extended to B_1 and B_2.

Abrams cites the case of two overlapping gradings as a flagrant case of particle interference. But overlapping does not necessarily decrease the average distance between particles in this size group to the point of particle interference.

Abrams cannot reconcile the statement that particle interference should account for both a larger water-cement ratio and a loss of strength by the water-cement law. Any loss of strength from particle interference is due to the effect on the cement paste in one of three ways: (1) Particle interference may stiffen the mix so that more mixing water is required, and dilutes the paste. (2)

Overdry mixtures, as described in the paper, are weak because the cement gel can form only at points of contact, the water being concentrated at these points by surface tension. Particle interference increases the effect of overdryness. (3) In wet mixtures particle interference forms large local voids in which the cement stratifies more readily. It was formerly believed by the author that particle interference was always accompanied by a loss of strength, but Powers has shown (11) that this is not necessarily the case with mortars rich in cement, as rich pastes of properly graded cement have sufficient cohesion to resist stratification. If the discontinuity is due to a high degree of particle interference, even rich mixtures will stratify.

TABLE I.—CEMENT SPACING IN NEAT PASTES AND MORTARS.

Data from Bulletin 137 (2)

Relative Water Content	$a\ c$	v/c	$\frac{c}{(v+c)}$	Water-Cement Ratio	Apparent Variation in Packing of Cement	
NEAT CEMENT PASTES						
1.00	0	0.82	0.550	0.35	100	100
1.20	0	0.92	0.520	0.44	95	100
1.40	0	1.24	0.447	0.60	81	100
CEMENT PASTE IN MORTARS WITH SAND NO. 6						
1.00	3.60	2.02	0.332	0.81	60	60
1.20	3.60	2.19	0.313	0.99	57	60
1.40	3.60	2.59	0.278	1.23	51	62

$\frac{c}{(v+c)}$ gives average density of cement paste and measures the packing of cement particles.

The cement had a normal consistency of 23 per cent.

Sand No. 6 was chosen because it is practically free from particle interference.

The concept of boundary effect does not seem to Mr. Abrams to agree with observed facts. The author could offer a mass of data which supports this concept when properly interpreted. One typical case is offered in the accompanying Table I. If there is no boundary effect produced by adding sand to cement paste, the increase in water and air voids must take place uniformly between the cement grains and result in their separation which makes a cement paste of lower density. A relative water content of 1.00 indicates the condition of maximum packing of particles. The spacing of cement grains, given in the table under the cement-space ratio, $c/(v + c)$, is computed for all the space in the mortar outside the sand. This average packing of cement grains in the mortars varies from 60 to 62 per cent of that in neat pastes for the same tamping and relative water content. The author knows of no reason why cement particles should be held further apart within the paste in mortars than in neat cement paste with equal compaction and consistency except for particle interference in the sand. Sand No. 6 used in this example is practically free from this. Viscosity or resistance to internal shear in cement paste will not serve as an explanation since it is evident from the accompanying Fig. 2 that relatively large increases of heavy aggregate mixed in mortar do not require added mixing water to overcome increased viscosity. A reasonable ex-

TABLE II.—DATA OF FIG. 13.

	Mix No. 1	Mix No. 2
Particle interference	none	much
Proportions:		
Cement, lb	94	94
Sand, lb	235	235
Gravel, lb	368	368
Fineness modulus	5.59	5.49
Real mix (Abrams)	4.77	4.83
Maximum size, in. (Abrams)	1½	¾
Tabular fineness modulus	5.85	5.03
Deduction (Angularity)	0.25	0.25
Allowable fineness modulus	5.60	4.78
Abrams' criterion	OK	Too coarse
Relative densities:		
¾ to 1½ in	0.178	0.103
⅜ to ¾ in	0.179	0.234
No. 4 to ⅜ in	0.179	0.124
Weymouth's criterion[a]	OK	Too harsh

Gravel is sub-angular.

Gradings of aggregates, No. 200 to 1½ in. inclusive.

Specific gravities, sand and gravel, 2.63.

Mixes hypothetical, only, to illustrate the figure.

[a] See Table II.

planation of these data point to the presence of boundary effect as described in the paper.

In answer to Abrams' request, the data for the mixes of Fig. 13 are given in the accompanying Table II. It appears that when judged by Abrams' method or by particle interference, mix No. 1 is equally acceptable while mix No. 2 is classified as poor by both. There are many cases, however, in which the limits established for fineness modulus can be shown to be too high when they are analyzed for particle interference.

With reference to the 6-in. slump used to illustrate the principles of the paper, other consistencies could be chosen equally as well as was stated in the paper, but any consistency chosen must be maintained throughout any single mortar-voids curve. Abrams does not state whether he approves the dry mixtures to which he calls attention, but the author decidedly does not favor them. Particle interference causes more air to become incorporated and renders the mixes still dryer, decreasing strength and increasing permeability. Usually the cost of handling dry concrete with equipment not specially designed for this class of work is increased much more than the saving in cement amounts to. The vibrator is used only after the concrete is brought to its final position.

With reference to 0.1-mm. differences computed for t, the factor of D in the formula is not sensitive to small variations in the sieve analysis; the number of decimal places shown for t is the same as given in tabular values for the wire openings.

The author cannot agree with Mr. Abrams to disregard the structural air in cement mixtures unless it can be shown that inundated concretes are plastic and provide sufficient temporary support to heavy sizes of aggregate to insure workability during placing operations. All the evidence points otherwise.

Mr. Anderegg compares the author's work with the method of grading he has worked out on an entirely different theory—that of starting with large pieces of aggregate and filling the voids to the highest degree possible to reduce the amount of cement required. His gap grading he admits requires special placing methods because of its harshness, but his continuous grading (based on the ratio 1.2) has excellent workability and low water-cement ratio for the consistency. Examination of this type of grading shows it to be practically free from particle interference. Anderegg in his original paper (6) pointed out the disastrous effect of "humps" in the grading, which reduced the strength of mortars 34 to 65 per cent. The author believes this was the first publication calling attention to the loss of strength due to an excess in one size group.

Mr. Carlson describes three tests in which the addition of an excess in one size group raised the water-cement ratio from 3 to 6 per cent. Differences in strength were about what would be expected from the water-cement ratios. He concludes that the effect of particle interference cannot be so great as has been concluded from tests on mortars.

These values for increase in mixing water seem small in comparison with those shown in the accompanying Fig. 2. A large amount of data is available to confirm the water variation trend of these figures. Without detailed data of Carlson's tests, the author cannot judge the degree of particle interference nor place it relatively in these diagrams.

Mr. Gilkey has contributed a very interesting discussion of the relation of mortar to concrete. But the author cannot wholly agree with his statement, that "the strength of a workable concrete is not usually, and probably never is, the strength of its mortar." While it cannot be denied that a large portion of published data confirms his view, it is nevertheless true that such test data contain concretes which would fall below line *P-P*, Fig. 2, and hence are undersanded from a particle interference point of view. The paper has shown that a cement paste cannot be wholly classified by its water-cement ratio and that its structure likewise must be considered. For example, the paste in the 1:2:4 mix in Fig. 2 has 6.1 gal. per sack and its structure is modified by the particle interference in the concrete. If it were possible to extract all coarse aggregate particles without removing any water, sand, or cement, the resulting mortar would have a consistency much greater than 1.1 which is the consistency of the diagram. With the cement paste more uniformly mixed throughout the mortar than was possible with the particle interference introduced in it by the coarse aggregate, cement stratification will be reduced and the strength of the mortar should be expected to be greater than it is in the concrete mixture. The 1:2 mortar mixed alone to 1.1 relative consistency requires only 5.1 gal. per sack. The 1.0 gal. difference explains why the mortar of poorly graded concrete has a too wet appearance.

Mr. Kellerman asks whether the mineral nature of the aggregate can be disregarded. In his work the author takes into account the shape of aggregates as well as size and grading. But differences due to mineral composition are like differences between different brands of cement, hardly to be covered by rules supposed to apply to all aggregates.

The author is sincerely grateful to Mr. Powers for his reply to a question of Mr. Kellerman stating that investigation of an aggregate for particle interference is a method of diagnosis rather than a method of design. In further reply, the author considers the mortar as a base to which coarse aggregate may be added and changed in size and proportion from time to time as pouring conditions make it necessary. So long as no particle interference is introduced into the mix and so long as other things are equal such as shape, texture of surface, soundness, etc., he considers that these changes can be made without fear of variation in consistency with the same water-cement ratio.

Mr. Merriman points out that the better the cement, the better the concrete. It was stated in the paper that the mortar-voids curve could show the quality of a cement in comparison with other cements. The author has made mortar-voids tests which indicated that the cement used could be improved. Changes to a logarithmic grading were made, which resulted in a very definite improvement in the quality of the concrete, and this accords with Mr. Anderegg's conclusions given in his criticism of this paper.

Mr. Powers describes investigations of particle interference made with the addition of powered silica to the mix in which very little effect of particle interference was observed. While the author has not seen the details of these data, this work seems to be another example of lessened cement stratification due to the cohesion provided by the powered silica.

In Powers' discussion of the author's theories elsewhere (11), he has suggested that $t = D_1$ is not necessarily the critical limit in the spacing of a dominant size group. Since differences in strength *per se* are not criteria for the presence of particle interference, its point of occurrence must be found where it increases voids in the matrix of the dominant size group. For coarse aggregates this point is readily determined. In mortars, however, the presence of boundary voids complicates the problem, and methods for locating the presence of particle interference given in the paper must be used. The author hopes the new series of tests being conducted by Mr. Powers will indicate definitely the relation of t to D_1, at which voids in the paste due to particle interference begin to increase in mortars.

Powers, like Abrams, is not willing to accept the theory of boundary voids. In the discussion of the data in the accompanying Table I, the author proves the presence of boundary effect without resort to a consideration of hexagonal close or cubical packing. The author has not sufficient data on tests with cement flour for computation of the spacing of its average particle in paste mixed with and without coarser cement sizes to determine whether cubical packing is probable.

Richart approached the interpretation of mortar-voids curves by theoretically filling the voids of sand with cement paste while the sand grains were virtually in contact. The author, in a similar study, found that particles of sized sands were separated in their mortars by the thickness of the coarsest grains in cement at their closest packing, and that the particles of the dominant group of graded sands likewise were separated by the thickness of the next smaller particles plus twice the thickness of the cement film, in basic mixtures. This separation of sand grains at basic mix by a constant thickness of film solved a puzzle with reference to sized sands. It is well known that the voids of dry-rodded size groups do not change in volume with the size of the average particle, yet in mortars the smallest diameters give the greatest voids at the point of minimum voids on the curve. The reason for this difference between the dry and mortar conditions was seen to be in the greater percentage of dispersion of small grains than large by the same separation of their adjacent surfaces due to the films of cement. The dry voids of sands have little relation to the voids of their mortars.

MR. SAMUEL S. STAHL[30] (*by letter*).—For more than four years this laboratory has made use of the study of relative densities of the size groups of the aggregates, according to Mr. Weymouth's method, in connection with determining the best grading of the aggregates in concrete; in most cases, concretes without particle interference have been recommended.

The practical results have been very good. Compressive strength of the concretes has been uniform and satisfactory as evidenced by more than ten thousand test cylinders and cores. Architects, contractors, engineers, and foremen have been pleased with the workability and the surface appearance after the forms are stripped.

There has been some adverse criticism of our concretes, that they are too cohesive, and flow sluggishly; to some observers they appear oversanded or deficient in the larger rock sizes. There has been no dissatisfaction about the compressive strength as related to the cement content.

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NOTE.—The numbers allotted, and which are used throughout the discussion, are in sequence with those used by the author.

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SOME FACTORS INFLUENCING THE BOND BETWEEN CONCRETE AND REINFORCING STEEL

By Raymond E. Davis,[1] Elwood H. Brown,[2] and J. W. Kelly[2]

Synopsis

Test results are presented which show that there is apparently no consistent relationship between bond strength and concrete compressive strength, but which seem to demonstrate that bond strength is largely influenced by the homogeneity of the paste in contact with the bar and by volumetric changes within the paste of the concrete surrounding the bar.

It is shown that bond strength varies greatly with type of cement; is less at initial slip for a rich concrete than a lean one; is less for horizontal than for vertical bars; is less for round than for square bars placed horizontally; may be greatly increased either by delayed vibration or by jigging during the setting period of the cement; is greatly decreased by repetitions of freezing and thawing or of wetting and drying; is greater for dry concrete than for saturated concrete; and under a variety of conditions is substantially greater for cements ground with TDA than for corresponding untreated cements.

Introduction

The character and quality of the cement paste, the manner in which it sets and hardens, and the conditions to which it is subjected not only during the setting period but also during the hardening period and at later ages perhaps exert a much more important influence upon the bond between concrete and reinforcing steel than upon the compressive strength of the concrete.

Concepts of Bond Action:

When water is added to cement and the process of hydration begins, each cement grain becomes surrounded with a supersaturated solution of hydrated compounds. This solution coagulates to form a gel. Thus each grain forms the nucleus of a gelatinous structure. Where the cement grains are sufficiently close to one another this gel structure soon becomes continuous so that, through the gel, the grains become knitted one to another, with intervening spaces filled with water.

During the early formation of this gel structure, there occurs a decrease in the total volume of the plastic cement paste. In other words, water and cement in a partially combined state occupy less absolute volume than in their separate states. This decrease in volume may be observed by the settlement of the surface of a dilute cement paste in a glass graduate. In construction it makes itself manifest through early settlement of concrete in the forms, even where the aggregate is completely saturated and the forms are tight. At the same time the cement grains with the gel structure occupy a greater absolute volume than that occupied upon first being wetted.

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As hydration proceeds the gel becomes more and more rigid and the void space within the hardened cement paste becomes less and less as a state of equilibrium is approached. During this second or hardening state, if there is a large excess of water available the paste will tend to occupy a greater gross volume as well as absolute volume, but if the paste is subjected to drying conditions the water content of the gel will decrease even though hydration of the deeper layers of the cement grain may not cease, and the paste will occupy a lesser absolute and gross volume.

Because of the tendencies toward growth or shrinkage within the structure of the hardened paste, there must be developed differential stresses even though the paste mass itself may be free from external restraints; and in the mass of a concrete member in service, the differential stresses between paste and aggregate must be even more pronounced.

Adhesion versus Friction:

For smooth plain bars, bond is generally considered to consist of two parts—that which is produced by *adhesion* of the hardened paste and that which is produced by *friction* between bar and concrete.

There appears to be no way of determining the magnitude of the strength of adhesion under the conditions that exist along a reinforcing bar. Certainly it must be much less than is the adhesion between cement paste and aggregate. Perhaps in many reinforced concrete structures it is so low as to be of no large consequence. In any case it would seem that we must rely upon the frictional resistance and not upon adhesion, for once there has been differential movement between steel and concrete, adhesion is likely to be destroyed.

Frictional resistance may be measured by the strength in bond after some slight movement has taken place and adhesion is broken. It is of course influenced by surface irregularities of the bar, but it must be proportional to the lateral pressures which exist or are developed between steel and concrete. How are these pressures developed and what are the factors which influence their magnitude? Before slip has taken place they may be developed by the contraction of concrete surrounding the bar; after slip has taken place they may be produced by wedging action and hence are influenced by the irregularities or roughness of the surface of the bar and by the resistance of the cement paste to crushing as this wedging action takes place. In any case, other things being equal, the greater the tendency of the concrete to "hug" the steel the greater would be the resistance to initial slip; and for bars with surface irregularities the greater this "hugging" value and the greater the resistance of the paste to crushing, the less would be the slip required to develop a given bond strength.

Factors Influencing Bond Strength:

Optimum resistance to crushing would seem not only to require a paste which possesses high strength but also to require a paste structure which is homogeneous, that is, one in which there is no water gain and in which the particles of cement are not flocculated but are uniformly dispersed so that there is a maximum area of contact between reinforcing bar and surrounding paste.

It seems reasonable to suppose that when the paste is in the plastic state a decrease in volume results in a tendency to draw away from the aggregate and from the reinforcing steel, both of which are restrained from free movement. In other words, while a mass of concrete may and does to some extent shrink or settle when it is still in the plastic state, yet it is restrained by the forms, the reinforcing steel, and the aggregate, and for this reason the decrease in over-all volume of the concrete may be (and probably is) very much less than would be the decrease in volume of the cement paste. This would seem to explain the lack of bond along the under side of aggregate and of steel reinforcement, even when dry mixes are used for which there could have been no water gain.

TABLE I.—RELATION BETWEEN BOND AND COMPRESSIVE STRENGTH.

Cement Content, sacks per cu. yd.	Cement				Water-Cement Ratio, by weight	Compressive Strength, lb. per sq. in.[a]		Bond Strength, per cent of compressive strength[b]			
	Type	C_3S, per cent	C_3A, per cent	Specific Surface, sq. cm. per g.		28 da.	5 mo.	Initial Slip		Maximum	
								28 da.	5 mo.	28 da.	5 mo.
4.5	Low-heat	31	3	1730	0.74	1480	3130	12.8	10.1	13.9	11.7
	Normal	51	10	1490	0.75	1960	2450	9.7	10.4	11.2	12.9
	Modified	51	6	1810	0.73	2330	2820	8.6	8.3	9.2	9.2
	High-early-strength	62	6	2160	0.72	2840	2930	6.7	5.8	6.9	6.8
	Portland-puzzolan[c]	....	....	2080	0.74	3210	4140	6.1	4.0	6.5	6.5
	Average	....	....	....	0.74	2380	3090	8.7	7.7	9.5	9.4
7.0	Low-heat	31	3	1730	0.43	5600	7970	2.6	2.7	6.6	5.7
	Normal	51	10	1490	0.45	4340	5610	3.3	3.2	8.3	7.0
	Modified	51	6	1810	0.43	6000	7700	2.1	1.9	3.8	3.8
	High-early-strength	62	6	2160	0.43	5370	6330	3.3	3.2	7.2	6.9
	Portland-puzzolan[c]	....	....	2080	0.49	4340	5710	4.2	3.8	10.9	9.6
	Average	....	....	....	0.45	5130	6660	3.1	3.0	7.4	6.6

[a] 3 by 6-in. cylinder; concrete compacted by external (table) vibration (3600 r.p.m.) for 10 sec.

[b] ⅜-in. plain round bar horizontal in 6-in. cube; mill scale not removed but bar cleaned with gasoline; concrete compacted 10 sec. by means of 1-in. internal vibrator (9000 r.p.m.); pull-out test.

[c] 70 per cent normal portland cement, 30 per cent calcined shale.

NOTE.—0 to ¾-in. local gravel; slump 3½ to 4 in.; specimens standard-cured and tested damp, 4 specimens per condition

Also, when the paste has become rigid, it seems clear that any tendency to increase in volume will cause the surrounding concrete to draw away from the bar and there may exist lateral tension between steel and concrete until a stress has been developed equal to the strength of adhesion. And in the opposite sense, any contraction that takes place in the hardened cement paste may be expected to produce a tightening of the concrete about the embedded bar.

For reasons just mentioned, optimum frictional resistance to sliding would seem to suggest not only a homogeneous paste but also one which during the period of setting, when the concrete is in a plastic or semi-plastic state, will exhibit a minimum contraction, and subsequently when the paste has become rigid will exhibit a minimum expansion.

If bond is conceived to be influenced by the qualities and behavior of the cement paste that have just been discussed, then some of the factors and conditions which might be expected to influence the strength in bond are: type and fineness of cement, dispersing agents, richness of mix and water-cement ratio, surface condition and shape and position of reinforcing bar, manner in which the concrete is placed and compacted or consolidated, period during which compaction or consolidation is effected, curing conditions, and variations in moisture and temperature such as may be produced by weather.

TEST RESULTS

With the object of indicating the possible influence of these factors upon bond strength, there are presented the results of several exploratory investigations made during the past few years. In general, the bond tests were made on bars embedded in gravel-concrete prisms or cylinders. Except as shown, the bars were plain hot-rolled steel of medium grade, the tests were of the "pull-out" or "push-out" type, and initial or first slip was considered to be such as would produce a movement of 0.0001 in. at the end of the bar opposite to that at which the load was applied.

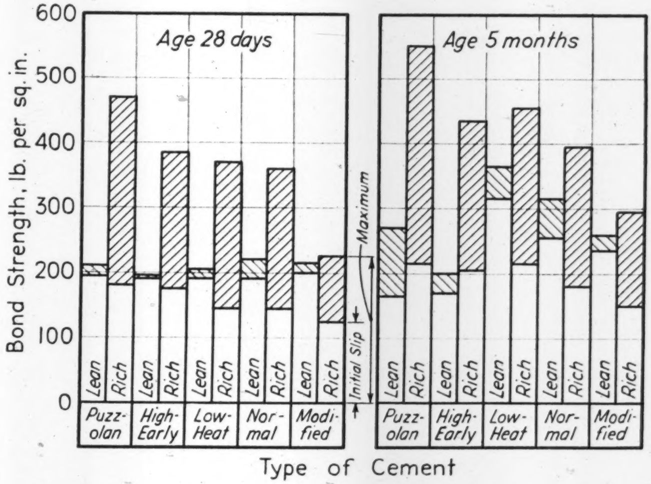

FIG. 1.—Effect of Type of Cement, Richness of Mix, and Age on Bond Strength. Data from Table I.

Relation Between Bond and Compressive Strength:

In Table I are given for five types of cement, two cement contents, and two ages of test, the bond strengths in terms of percentages of the compressive strengths. Considering bond strength at initial slip, it will be noted that while on the average the percentage for the mix containing 4½ sacks of cement per cu. yd. is approximately three times that for the mix containing 7 sacks per cu. yd., among the different types of cement this ratio varies from less than 1½ for the portland-puzzolan cement to approximately 5 for the low-heat portland cement. Considering the maximum bond, the ratio of lean mix percentage to rich mix percentage is about $\frac{2}{3}$ for the portland-puzzolan cement at one extreme to nearly 2½ for the modified portland cement at the other extreme.

For the lean mix, regardless of type of cement or age at time of test, generally the higher the compressive strength the lower the ratio of bond strength to compressive strength both at initial slip and at the maximum. For the rich mix there is not this consistent relationship, and it appears that the type of cement may to a marked degree influence the ratio of bond to compressive strength.

Effect of Type of Cement and Richness of Mix:

In order to show the effect of richness of mix upon the actual values of bond strength, there have been prepared for each of the several types of cement the diagrams of Fig. 1. The ordinates to the bottom of the cross-hatched areas represent bond strength at initial slip, and the ordinates to the top of the cross-hatched areas represent maximum bond strength. For the age of 28 days, it is seen that for all types of cement the bond strength at initial slip is *less* for the rich mix than for the lean mix, while the maximum bond strength is *greater* for the rich mix than for the lean mix. For the lean mix there is little difference between initial and maximum values, and among the various cements these values are nearly constant; for the rich mix there is a large difference between initial and maximum values for a given cement, and among the several types of cement the differences both in bond strength at initial slip and in maximum bond strength are large. The bond strengths are highest for the portland-puzzolan cement and lowest for the modified portland cement.

Perhaps the reason why bond strength at initial slip is lower for a rich than for a corresponding lean mix is that in the plastic state the greater quantity of cement paste in the rich mix contracts more and therefore tends to draw away from the embedded bar more than does the cement paste in the lean mix. That is, the contraction of a mixture during the plastic state, other things being equal, would be expected to be proportional to the paste content. That this must be substantially true is borne out by the results of experiments, by observations of the settlement of concrete in forms, and by the tendency of concrete surfaces to check during the setting period. Also, one may conjecture that during the hardening period under moist conditions the concrete of the rich mix will expand more than the concrete of the lean mix, and hence there will be a greater tendency towards separation of the concrete and the embedded bar.

From the small differences between the initial and maximum values for the lean mix and the relatively large differences between initial and maximum values for the rich mix, it seems evident that the maximum bond strength is in a large measure dependent upon the crushing strength of the cement paste under the wedging action that must take place when slip occurs. The fact that for the rich mix the maximum bond strength for the portland-puzzolan cement is so much higher than that for any of the other types of cement may perhaps be attributed to a more homogeneous paste structure. For the portland-puzzolan cement employed, it was observed that it produced a concrete mix which was plastic to the point of being sticky, with no tendency whatever towards water gain.

TABLE II.—EFFECT OF TDA.

Cement	Specific Surface of Cement, sq. cm. per g.		Compressive Strength, lb. per sq. in.		Maximum Bond Strength, lb. per sq. in.	
	Untreated	TDA	Untreated	TDA	Untreated	TDA
A	2280	2390	5340	6250	110	135
B	2250	2250	5380	4940	125	160
C	1830	1790	4800	5000	100	150
D	1710	1720	5130	5550	90	140
E	1425	1480	4400	4840	140	165

NOTE.—Cement-aggregate ratio, 1:5.0 by weight; water-cement ratio, 0.60 by weight; 0 to ⅜-in. gravel aggregate; ¾-in. plain round cold-rolled bar horizontal in 4-in. cube; concrete compacted by external vibration; specimens standard-cured, and tested damp at age 14 days; push-out test; 2 specimens per condition.

Figure 1 also shows corresponding diagrams of bond strengths at the age of 5 months. For the lean mix, the differences between initial and maximum bond strengths are greater at the later age than at the earlier age. This is also generally true for the rich mix.

From a study of the diagrams of Fig. 1, it would appear that for a lean mix and at the early age of 28 days the type of cement has comparatively small effect upon either the bond strength at initial slip or the maximum bond strength.

For the rich mix, however, it appears that the type of cement exercises a marked influence upon both the initial and the maximum strengths. At the later age of 5 months it appears that the magnitude of the bond strength both at initial slip and at the maximum may be considerably influenced by the type of cement.

Effect of TDA:

In Table II are given the maximum bond strengths for a group of portland cements representing a range of composition and fineness, ground with and without TDA, a dispersing agent.[3] The bars were cold-rolled steel, and the specimens were tested at the age of 14 days. It will be noted that in every case the bond strength is greater (average 32 per cent) for a cement ground with TDA than for the corresponding untreated cement, even though the treated cements are of substantially the same fineness as the corresponding untreated cements. The relative increase in bond strength due to the use of TDA is greater than the relative increase in compressive strength of concrete.

The improvement in bond produced by the use of TDA seems to be attributable primarily to the generally greater homogeneity of hardened cement pastes containing this compound. Microscopic studies indicate that this greater degree of homogeneity exists for cements ground with TDA, and it appears that its action must be to create a more complete dispersion of the cement grains throughout the paste during the period before setting occurs. Examination of the bond surfaces of concrete after test quite generally indicates that regardless of the consistency of the mix there existed a more uniform contact between bar and concrete for cements ground with TDA than for untreated cements. There is perhaps also the factor of closer gripping of the bar, since it has been observed at least for some cements ground with TDA that the expansion of mortar bars may be somewhat less during the period of moist curing than is the expansion of corresponding bars containing untreated cement.

TABLE III.—EFFECT OF SHAPE AND POSITION OF BAR AND TYPE OF TEST.

Bar		Slump, in.	Water-Cement Ratio, by weight		Compressive Strength, lb. per sq. in.[c]		Bond Strength, lb. per sq. in.			
							Initial Slip		Maximum	
Shape	Position		Untreated	TDA	Untreated	TDA	Untreated	TDA	Untreated	TDA
Round.......	Vertical[a]	3¼	0.50	0.49	4280	4810	215[d]	300[d]	495[d]	660[d]
							190	225	485	560
	Horizontal[b]	3¼	0.50	0.49	4280	4810	105	115	115	160
		6½	0.55	0.53	4100	4520	90	100	100	130
Square.......	Horizontal[b]	2½	0.48	0.47	4880	5680	120	135	275	325
		6	0.54	0.52	4330	5110	195	205	380	365

[a] Bond specimens 6 by 6-in. cylinders.
[b] Bond specimens 6-in. cubes.
[c] 3 by 6-in. cylinders.
[d] Push-out tests.

NOTE.—Cement-aggregate ratio, 1:5.6 by weight; 0 to ¾-in. local gravel; high-early-strength portland cement; ⅝-in. plain bar; concrete hand-tamped; specimens standard-cured and tested damp; test at age 7 days; pull-out tests except as noted by reference *d*; 4 specimens per condition.

Effect of Shape and Position of Bar and Type of Test:

The values of Table III show that the bond strengths at both initial slip and maximum are greater for vertical bars than for horizontal bars, are greater when compression is applied to the bar than when tension is applied to the bar, and are greater for square bars placed horizontally than for round bars placed horizontally. Where the bars are placed horizontally there is a greater difference in bond strength between round and square bars when the mix is wet than when it is dry; also, there is a much greater increase in strength between initial slip and maximum for square bars than for round bars.

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[3] TDA is a mixture of tri-ethanol amine salts and highly purified soluble calcium salts of modified lignin sulfonic acids.—ED.

TABLE IV.—EFFECT OF VIBRATION.

Method of Compacting Concrete	Time of Vibration, sec.	Bond Strength, lb. per sq. in.	
		Initial Slip	Maximum
Hand tamping	0	205	740
External vibration (molds clamped to vibrating table)[a]	15	210	845
Axial bar vibration (air hammer operated axially against top end of bar)[a]	30	330	1075

[a] Concrete hand-tamped; specimens vibrated immediately after molds were filled.

NOTE.—Cement content 1.50 bbl. per cu. yd.; water-cement ratio, 0.61 by weight; slump 4 in.; modified portland cement; 0 to ¾-in. local gravel; ⅞-in. round deformed bar vertical in 6 by 6-in. cylinder; specimens standard-cured, and tested damp at age 28 days; pull-out test; 3 specimens per condition.

It seems probable that these differences as between vertical and horizontal bars and as between round and square bars are due to the shrinkage and settlement of the cement paste while still in its plastic state. When the bars are placed horizontally this shrinkage tends to leave a void space beneath the bar. For the round bar this tendency may exist over substantially the lower half of the circumference; for the square bar it exists only over the lower quarter. Thus a larger percentage of the surface of the square bar may be considered as effective in bond. If there is water gain beneath the bar or a tendency of the cement paste to settle away from the bar, it seems clear that the round bar will be much more loosely gripped than will the square bar, and hence it is to be expected that the difference between bond strength at initial slip and maximum bond strength would be much less for a round bar than for a square bar.

It will be noted that the use of TDA quite generally increases the bond strength regardless of the position or shape of bar and regardless of the consistency of the mix. This again seems to point rather conclusively to a greater homogeneity of paste structure for cements ground with TDA than for corresponding untreated cements.

TABLE V.—EFFECT OF DELAYED VIBRATION.

Vibration	Bond Strength at 28 days						
	Kind	lb. per sq. in.	Percentage of Strength of Unvibrated Specimens				
		Unvibrated	Unvibrated	0 hr.	3 hr.	6 hr.	9 hr.
External (table)[a]	Initial slip	205	100	103	29	88	142
	Maximum	740	100	114	133	143	126
Bar[b]	Initial slip	205	100	...	200	190	190
	Maximum	740	100	...	147	155	141
Bar (axial)[c]	Initial slip	205	100	161	200	212	83
	Maximum	740	100	145	154	162	128

[a] Specimen molds clamped to table; table vibrated by means of electric form vibrator (3600 r.p.m.) for 15 sec.

[b] Shaft of 1-in. internal vibrator, horizontal, clamped to top end of vertical bar; vibration (7500 r.p.m.) for 30 sec.

[c] Air hammer operated axially (vertically) against top end of bar for 30 sec. (3000 r.p.m.).

NOTE.—Cement content 1.50 bbl. per cu. yd.; water-cement ratio, 0.61 by weight; slump 4 in.; modified portland cement; 0 to ¾-in. local gravel; ⅞ by 18-in. round deformed bar vertical in 6 by 6-in. cylinder; concrete hand-tamped; vibration at ages indicated; specimens standard-cured and tested damp; pull-out test; 3 specimens per condition.

Effect of Vibration:

In Table IV are given values of bond strength at initial slip and maximum, for three conditions of compaction. It will be noted that external vibration applied to molds clamped to a vibrating table increased the bond strengths but slightly over those obtained by hand tamping alone; on the other hand, when vibration through the use of an air hammer was applied axially to the end of the bar, the effect was to increase by about 50 per cent both the bond strength at initial slip and the maximum bond strength. The reason for this marked increase in bond strength is not clearly evident, but it seems possible that the vibration of the bar may have produced a remixing action of the cement paste in the immediate vicinity of the bar, and that through this remixing action there was obtained a more homogeneous paste structure at the contact surfaces. It should be noted that the bars were vertical. Whether the same relationship would hold for bars placed horizontally has not been determined.

TABLE VI.—EFFECT OF SUSTAINED JIGGING.

Concrete			Maximum Bond Strength at 28 Days							
Aggregate-Cement Ratio, by weight	Water-Cement Ratio, by weight	Maximum Size of Aggregate, in.	lb. per sq. in.	Percentage of Strength of Unjigged Specimens						
			0 hr.[a]	0 hr.[a]	½ hr.	1 hr.	2 hr.	3 hr.	4½ hr.	6 hr.
6.2........	0.58	1½	525	100	131	145	...	177	186	...
8.7........	0.60	¾	265	100	185	212	241	238	240	241

[a] Specimens not jigged.

NOTE.—1-in. plain round bar vertical in 6 by 6-in. cylinder reinforced with 4-in. helix of $\frac{3}{16}$-in. wire; normal portland cement; crushed aggregate; concrete hand-tamped; jigging table dropped ¼ in. 300 times per min., for periods indicated; standard curing 21 days, then storage in air of laboratory 7 days; specimens tested dry; pull-out test; 3 or 4 specimens per condition.

Effect of Delayed Vibration:

In Table V are given the bond strengths of specimens for which the concrete was placed by hand and the specimens vibrated at some later period. Deformed bars were employed. The conditions of vibration were as follows: (1) the specimen itself was vibrated by clamping the mold to a vibrating table, (2) the bar was vibrated by attaching to its upper end the shaft of a high-frequency internal vibrator, and (3) the bar was vibrated axially by a light air hammer operated against its upper end.

For the first condition of vibration, when the vibration was applied externally at either 3 or 6 hr. the bond strength at initial slip was less than for concrete not vibrated; but when the vibration was applied at the age of 9 hr. its effect was to increase by 42 per cent the bond strength at initial slip. For all ages at which vibration was applied, the effect was to increase the maximum bond strength substantially.

For the second condition, with the bar vibrated at high frequency but with relatively small amplitude, the effect was to increase the bond strength at initial slip by about 100 per cent and to increase the maximum bond strength by roughly 50 per cent.

For the third condition, in which the bar was vibrated axially with considerable force, when the vibration was applied at ages of either 3 or 6 hr. the effect was to increase the bond strength at initial slip approximately 100 per cent; but when the vibration was applied at the age of 9 hr. the bond strength at initial slip was lower than for the specimens to which no vibration was applied. When the age at which vibration was applied was 6 hr. or less, the maximum bond strength was increased approximately 50 per cent.

It appears that the effect of bar vibration after the cement has taken its initial set may be to reconsolidate the concrete in the vicinity of the bar as well as to produce a more homogeneous structure for the paste surfaces in contact with the bar. External vibration by itself would be sufficient to reconsolidate the mass and hence to relieve any internal strains that may be set up through shrinkage of the paste, but would perhaps produce no remixing action along the bar.

The results of these tests, which have been qualitatively verified by other tests not here reported, would seem to point to the general desirability of vibrating the vertical steel in reinforced concrete members such as columns at some time between initial and final setting of the cement. They perhaps indicate the desirabillty of delayed vibration of the steel regardless of its position.

Effect of Sustained Jigging:

In Table VI is shown the effect of sustained jigging on maximum bond strength. For these tests the specimens were rigidly held to a jigging table for which the motion was vertical. The table dropped $\frac{1}{4}$ in. at the rate of 300 drops per min. The bars were vertical. The concrete for the specimens was hand tamped. Immediately thereafter the operation of jigging was begun, and it was continuous up to the ages indicated in the table.

For the leaner mix, the increase in bond strength due to jigging was in excess of 100 per cent for periods of jigging between 1 and 6 hr. There was no considerable effect upon bond when the period of jigging was in excess of 2 hr. For the richer mix the increase in bond strength due to jigging was less pronounced.

These large increases in bond strength through jigging seem to be explained by the continued consolidation or compacting effect of the jigging operation. During the period when the cement paste is in a plastic or semi-plastic state and is undergoing volumetric shrinkage, the jigging offsets the shrinkage and causes the paste to grip the bar more tightly.

Effect of Condition of Storage:

In Table VII are given the results of tests to determine the effect of air-drying upon bond strength. For series I, where the tests were made at the age of 3 months, it is seen that the bond strength at initial slip is nearly proportional to the compressive strength of the concrete; that is, it is least for the concrete cured only one day moist and for which the compressive strength is also the least, and is greatest for the concrete cured 7 days moist for which the compressive strength is also the greatest. However, regardless of the duration of moist curing, the maximum bond strength for air-stored specimens is greater than that for the specimens maintained continuously moist. With regard to maximum bond strength, results of the tests of series II exhibit the same trend both for the untreated cement and for the corresponding cement ground with TDA.

The results of these tests would make it appear that while the bond strength at initial slip is not appreciably affected by such shrinkage as has taken place in the concrete during the drying period,

TABLE VII.—EFFECT OF CONDITION OF STORAGE.

	Age at Test, days	Moist-Curing Period, days	Dry-Storage Period, days	Condition at Test	Compressive Strength, lb. per sq. in.		Bond Strength, lb. per sq. in.		
							Initial Slip	Maximum	
					Untreated	TDA	Untreated	Untreated	TDA
Series I[a].....	91	91	...	Damp	4780	....	125	190	...
		1	90	Dry	3090	....	90	260	...
		7	84	Dry	4980	....	150	290	...
Series II[b]....	28	28	....	Damp	6060	7720	....	155	170
		1	27	Dry	4500	4620	....	215	285

[a] Cement-aggregate ratio, 1:5.6 by weight; water-cement ratio, 0.48 by weight; slump 4 in.; modified portland cement; 0 to ¾-in. local gravel; compression specimen 3 by 6-in. cylinder, with concrete compacted by external vibration for 16 sec.; bond specimen 6-in. cube, with concrete compacted by 1-in. internal vibrator (9000 r.p.m.) for 8 sec.; ⅝-in. plain round bar, horizontal; storage at 70 F.; pull-out test; 4 specimens per condition.

[b] Cement-aggregate ratio, 1:5.0 by weight; water-cement ratio, 0.60 by weight; 0 to ⅜-in. gravel; compression and bond specimens 4-in. cubes, with concrete hand-tamped; ¾-in. plain round cold-rolled bar, horizontal; storage at 70 F.; push-out test; 2 specimens per condition.

the effect of this shrinkage is to increase the maximum bond strength materially.

Effect of Temperature of Dry Storage:

When after a preliminary period of moist curing the temperature of reinforced concrete is raised, it may be reasoned that there would be a tightening of the concrete about the embedded steel because of the greater tendency towards expansion of the steel. The pressures that might thus be developed would depend upon the difference between thermal expansions of steel and concrete and the yielding characteristics of the concrete; and, other things being equal, they would be less if the temperature increase occurred at an early age while the concrete was still relatively plastic than if this increase occurred at a later age when the concrete was relatively non-yielding. For similar reasons, a drop in temperature might be expected to result in a lessening in pressure between concrete and embedded steel.

TABLE VIII.—EFFECT OF TEMPERATURE OF DRY STORAGE.

Moist-Curing Period at 70 F., days	Dry-Storage Period, days	Temperature of Dry Storage and Test, deg. Fahr.	Compressive Strength, lb. per sq. in.[a]	Bond Strength, lb. per sq. in.[b]	
				Initial Slip	Maximum
7	84	40	4420	100	210
7	84	70	4980	150	290
7	84	100	4280	105	240
84	7	100	5400	165	285

[a] 3 by 6-in. cylinder; concrete compacted by external (table) vibration (3600 r.p.m.) for 10 sec.

[b] $\frac{5}{8}$-in. plain round bar horizontal in 6-in. cube; bar wire-brushed; concrete compacted by means of 1-in. internal vibrator (9000 r.p.m.) until film of water appeared at surface (normally 8 sec.); pull-out test.

NOTE.—Cement-aggregate ratio, 1:5.6 by weight; water-cement ratio, 0.48 by weight; slump 4 in.; modified portland cement; 0 to $\frac{3}{4}$-in. local gravel; specimens tested dry at age 3 months. Coefficient of thermal expansion of concrete 0.0000045 per deg. Fahr.; 4 specimens per condition.

The results of tests shown in Table VIII indicate that the bond strength, as well as the compressive strength, may be substantially affected by either an increase or decrease in temperature. Whatever may be the factors contributing to these differences in bond strength, it is not apparent that the differential thermal expansion between steel and concrete plays an important part. The tests are not sufficiently comprehensive to justify any conclusion, but the decreases in bond strength due either to a raising or lowering of temperature are sufficiently large to point to the desirability of more extensive investigations of temperature effects.

TABLE IX.—EFFECT OF TREATMENTS SIMULATING THE ACTION OF WEATHER.

Cyclic Treatment	Number of Cycles	Temperature at Test, deg. Fahr.	Compressive Strength, lb. per sq. in.[a]	Bond Strength, lb. per sq. in.[b]	
				Initial Slip	Maximum
None (continuously in fog at 70 F.)	0	70	4780	125	190
Freezing and thawing (saturated; 16 hr. at 0 F., 8 hr. at 100 F.)	56	100	3960	10	150
Wetting and drying (8 hr. in water at 70 F., 16 hr. in air blast at 160 F.)	56	70	4540	10	120

[a] 3 by 6-in. cylinder; concrete compacted by external (table) vibration (3600 r.p.m.) for 10 sec.

[b] $\frac{5}{8}$-in. plain round bar horizontal in 6-in. cube; bar wire-brushed; concrete compacted by means of 1-in. internal vibrator (9000 r.p.m.) until film of water appeared at surface (normally 8 sec.); pull-out test.

NOTE.—Cement-aggregate ratio, 1:5.6 by weight; water-cement ratio, 0.48 by weight; slump 4 in.; modified portland cement; 0 to $\frac{3}{4}$-in. local gravel; treated specimens standard-cured to 28 days, then subjected to cyclic treatment; specimens tested damp at age 3 months; 4 specimens per condition.

Effect of Weathering Conditions:

In Table IX comparison is made between bond specimens which have been continuously moist-cured and those which have been subjected to 56 cycles of (1) freezing and thawing or (2) wetting and drying.

Either of these treatments reduces the bond strength at initial slip to a negligible quantity and substantially reduces the maximum bond strength. It has been observed that repetitions of freezing and thawing or wetting and drying quite generally result in a marked expansion of concrete, which perhaps indicates a breaking of the adhesive bond between aggregate and cement paste. It seems reasonable to suppose that similar action would take place between embedded steel and surrounding concrete, and that the effect would be not only to destroy adhesion between bar and paste but also greatly to reduce the frictional resistance developed at initial slip.

TABLE X.—EFFECT OF SUSTAINED STRESS.

Sustained Stress (Maintained from 7 days to 3 months)		Compressive Strength, lb. per sq. in.[c]	Bond Strength, lb. per sq. in.[d]	
Type	lb. per sq. in.		Initial Slip	Maximum
None	0	4780	125	190
Bond (applied to bar)[a]	100	4780	155	330
Compressive (applied to top and bottom faces of cube)[b]	600	4780	130	345

[a] See Fig. 2(*a*); sustained stress released just prior to pull-out test.

[b] See Fig. 2(*b*); sustained compressive stress applied by means of an assembly of rods, plates, and coiled spring; stress maintained during pull-out test.

[c] 3 by 6-in. cylinder, concrete compacted by external (table) vibration (3600 r.p.m.) for 10 sec.

[d] ⅝-in. plain round bar horizontal in 6-in cube; bar wire-brushed; concrete compacted by means of 1-in. internal vibrator (9000 r.p.m.) until film of water appeated at surface (normally 8 sec.); pull-out test.

NOTE.—Cement-aggregate ratio, 1:5.6 by weight; water-cement ratio, 0.48 by weight; slump 4 in.; modified portland cement; 0 to ¾-in. local gravel; specimens standard-cured, and tested damp at age 3 months; 4 specimens per condition.

Effect of Sustained Stress:

The effect of plastic flow or time yield of concrete upon bond strength is deserving of some consideration. Two cases are here considered: (1) The case where bond stress somewhat less than the strength at initial slip is sustained for a considerable period before test, paralleling the conditions in a beam where there is continuously present the bond stress due to dead load, which stress may at any time be increased by live load; and (2) the case where there is sustained in the concrete for a considerable period a compressive stress acting normal to the axis of the bar, paralleling conditions which are found, say, in the beams at the columns of a building frame.

In Table X comparison is made between the bond strength of specimens subjected to each of these conditions and that of specimens to which no load was applied prior to the time of testing. It appears that either sustained bond stress or a sustained compressive stress normal to the axis of the bar slightly increases the bond strength at initial slip and considerably increases the maximum bond strength.

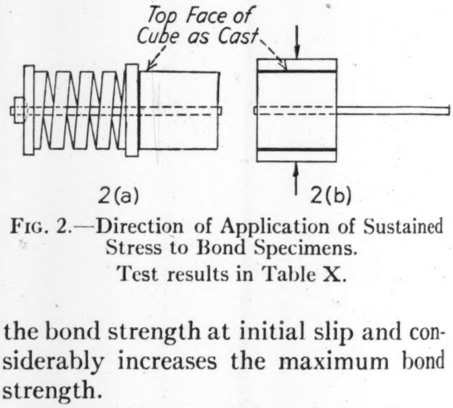

FIG. 2.—Direction of Application of Sustained Stress to Bond Specimens.
Test results in Table X.

It should be noted that the specimens were stored and tested moist. Under these conditions, during the period of storage there would occur some expansion of the concrete which would tend to reduce the frictional resistance of the bar to sliding; and plastic flow in either bond or compression might be effective in increasing bond strength. In the more usual case where the concrete is subjected to drying, and due to drying shrinkage the frictional resistance is ordinarily increased, the effect of sustained load upon the bond strength might be inappreciable.

CONCLUDING STATEMENT

The tests herein reported have shown the same general lack of correlation between compressive strength and bond strength as that observed by other investigators. It appears that the *compressive* strength of concrete is largely determined by the strength of the hardened cement paste and its adhesion to the aggregate. The results of the tests here presented indicate that *bond* strength between concrete and reinforcing steel of given roughness depends in part on paste strength after initial slip has occurred but may be largely determined by the character of the cement paste, its volumetric behavior as it sets and hardens, and the conditions to which the paste is subjected not only as it sets and hardens but also at later ages.

The generally close relations between bond strength and paste characteristics or manner of paste treatment lend support to the view that the bond strength is dependent upon the magnitude and uniformity of lateral pressures that exist or may be developed between steel and surrounding concrete. The uniformity of pressure seems to be governed by the homogeneity and uniformity of the paste structure surrounding the steel. The tightness with which the steel is gripped by the surrounding concrete, while dependent upon paste strength and rigidity, may often be more dependent upon the tendency of the paste to shrink away from the steel during the setting period and subsequently upon the tendency of the paste to expand or contract with moisture and thermal changes.

While the tests here reported are perhaps not sufficiently comprehensive to justify the drawing of definite conclusions, the results would seem to indicate that:

1. Bond strength varies greatly with the type of cement.
2. At initial slip, bond strength is generally lower for the rich mix than for the lean one.
3. Bond strength is less influenced by type of cement in the lean mix than in the rich mix.
4. With the rich mix, under the condition of moist curing the maximum bond strength is nearly twice as great for the portland-puzzolan cement as for the modified portland cement.
5. Bond strength is substantially higher for the cements ground with the dispersing agent TDA than for corresponding cements not so treated.
6. Bond strength is less for horizontal bars than for vertical bars.
7. Bond strength is less for round horizontal bars than for square horizontal bars.
8. For vertical bars, bond strength may be greatly increased by vibrating the bar at some time between initial and final setting of the cement. This treatment doubles the strength at initial slip and increases the maximum bond by 50 per cent.
9. Bond strength is increased more by delayed vibration of the bar than by delayed external vibration of the concrete.
10. For vertical bars, bond strength is greatly increased by continuous jigging during the setting period.
11. Bond strength is greater for concrete subjected to drying than for saturated concrete.
12. Bond strength is greatly reduced by freezing and thawing or by wetting and drying with alternations of high and low temperature.

Consideration of the wide departures from any fixed relationship between bond strength and compressive strength of concretes such as are now commonly employed in construction, and consideration of those factors which may have little effect on compressive strength but a large effect on bond strength, would make it appear questionable whether the working stress in bond should be based solely on the compressive strength and would point to the desirability of basing the working stress on the results of a standard bond test.

Where bond is a matter of importance, the results of the tests seem to point to the desirability of specifications which will so control the selection of materials, the design of mixes, and the operations of concrete placement and compaction that conditions for high bond strength—such as paste homogeneity and uniformly tight contact between concrete and steel—will be most favorable.

The fact that by alternations of freezing and thawing or wetting and drying the bond strength may be seriously impaired while the compressive strength may be affected only to a minor degree suggests a lower working stress in bond for members exposed to weather or to large variations in temperature than for those members which are protected.

Acknowledgments:

The authors desire to acknowledge the cooperation of Harmer E. Davis, Assistant Professor of Civil Engineering; E. J. Garbarini, N. E. Haavik, and J. F. Ryan, members of the staff of the Engineering Materials Laboratory; and R. H. Burger, Anatoli Eremin, V. L. Hanson, L. G. Horton, O. W. Schrader, J. G. Standley, J. G. Sutton, L. L. Thomas, and E. M. Westall, former undergraduate or graduate students in Civil Engineering; all of the University of California.

DISCUSSION

Mr. E. Viens.[1]—I should like to ask Mr. Davis what he means by lean and rich mixes. Were the aggregates the same for all tests, that is, maximum size?

Mr. Raymond E. Davis.[2]—The maximum size of aggregate was three-quarters of an inch.

The aggregate was from the same source and the grading was the same for the rich mixes as for the lean ones.

Messrs. H. J. Gilkey,[3] S. J. Chamberlin,[3] and R. W. Beal[3] *(by letter)*.—This paper constitutes one of several recently published[4] which venture into the field of bond. The writers know of several other investigations in progress. Some of these should see publication relatively soon.

This revival of interest in bond is timely, for indeed there have been many changes in both the science and the art of concrete since the pioneer work of Withey and the classic work of Abrams. For a long time there have been few changes in bond concepts and design practices, but there have been mounting doubts concerning the validity of some of these concepts and practices as applied to present-day conditions.

The authors' tests, frankly of an exploratory nature, constitute a useful supplement to the other tests recently completed and in progress; for, in a number of respects, they tap relatively new variables and aspects of bond. Where there are overlappings, these, too, are to the good, either as verification or as points calling for further study.

The speculative portions of the paper discuss a number of interesting questions to which no one is likely to have factual answers for some time. Questions of the extent to which adhesive resistance to sliding resembles or differs from frictional resistance, and whether or not surface roughness differs in any fundamental respect from lug action are always tantalizing. Is the so-called law of friction, as treated in mechanics, a fundamental thing, or simply an empirical relationship observed to hold true for surfaces with textures fine enough to permit sliding rather than locking?

The writers and some of the other current investigators have been devoting considerable attention to the progressive nature of bond action, which brings out the important fact that computed bond resistances of pull-out specimens will differ greatly for different lengths of embedment, since the maximum resistance is at first always concentrated near the point of entry on the loaded side. There must be a progressive slippage or successive letting go along the bar before either load or slip is transmitted to the unloaded end where "first slip" has always been measured. F. R. McMillan, in discussing the so-called "first slip" aptly terms it the "last slip" since it is observed at the last point on the bar to have slipped.

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[2] Professor of Civil Engineering, University of California, Berkeley, Calif.

[3] Head, Assistant Professor, and Instructor, respectively, Department of Theoretical and Applied Mechanics, Iowa State College, Ames, Iowa.

[4] In the September, 1938, *Journal* of the American Concrete Inst., Vol. 1, No. 1, pp. 1-20, the writers in their paper on "The Bond Between Concrete and Steel," outline briefly some of these developments and list or refer to lists which include the more important of the publications on bond which have come to their attention.

Because of the inequality of bond stress along the bar, the length of embedment (of the bar) must be recognized as one of the significant variables when bond tests are compared. Moreover, we need to shift our attention to the other end of our pull-out (or push-out) specimen and find out just what does happen at the loaded end and why.

In several places are noted such statements as: "... even when dry mixes were used for which there could have been no water gain." The writers question whether there can be such a thing as a properly placed concrete that was free from water gain. They know of no better or dependable criterion for adequate placement than the water sheen which appears at the surface of any freshly deposited concrete just as soon as capillary continuity has been established throughout the mass. Until the sheen appears, placement is incomplete for there are discontinuities between the surface concrete and that below. Yet, that sheen is simply a film of water which will be present between the underside of the bar (and aggregate particles, large and small) and the mortar bed below. The water or air bubbles can rise no farther and will in all probability act as a weakening layer or stratum. It is true, of course, that the wetter the mix, the thicker will the water film or layer become. The adherence of the aggregate of horizontally fractured concrete to the upper portion of the block as cast holds true, in the writers' observation, for wet and dry consistencies alike, provided only that they were not crumbly as placed. Observations include those on zero slump concrete vibrated into position. When the bar, or the aggregate, is free to follow down, as the mortar shrinks, the water gain will be less injurious than if not, for the discontinuity will be thinner than otherwise. When there is considerable mortar shrinkage, combined with inability of aggregate to follow down, actual caves may result from water gain, and it is these that the authors were evidently visualizing. They are more in the nature of honeycomb, although both elements are present.

The variation of bond resistance with type of cement is not surprising in the light of Glanville's tests.[5] Glanville secured much higher bond stresses from alumina cement than from portland cement. He ascribed the difference to the greater shrinkage of the alumina cement in curing, which produced normal pressure on the bars, thereby increasing the frictional resistance to sliding. In view of the relatively large volume change which characterizes alumina cement, the explanation is at least tenable. Whether or not differences in the shrinkage characteristics of the cements were important in the different bonds developed in the authors' tests is an interesting question.

Limited reconnaissance pull-out tests reported in Vol. 16 of the *Proceedings* of the Highway Research Board p. 90 (1936), tend to confirm the authors' conclusion relative to the greater bond resistance of concrete dry at test over that in the saturated condition. If this apparent increase is valid and is due, as the authors imply, to shrinkage from drying, it should be possible to verify the indication by tests on pull-outs in blocks of different diameters. The thicker the shrinking mass around the bar the greater should be the pressure exerted upon the bar. For $\frac{1}{2}$-in. bars embedded 6 in., Glanville found only a slight increase in bond resistance for 28-day, air-cured, alumina concrete, as block diameter was increased from 3 to

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[5] W. H. Glanville, "Studies in Reinforced Concrete. I Bond Resistance," *Building Research Paper No. 10*, Department of Scientific and Industrial Research, London, p. 6 (1930).

7 in. Perhaps the element of lubrication by the moisture is a significant factor as well as, or instead of, that of lateral shrinkage.

The writers' tests do not support the indication that initial slip occurs at a lower unit bond stress for rich mixtures than for lean ones. The Highway Research Board tests show no great difference, but where one did exist it was invariably in favor of the richer mixture.

The data on vibration are a splendid supplement to and extension of the work of Withey reported in the 1936 (Vol. 16) *Proceedings* of the Highway Research Board. The evidence on this point seems to be such as to allay all fears about the possibility of vibration "jarring the bars loose" from the concrete. Not only that, but the reworking or remixing action from delayed vibration is seemingly a splendid antidote for the inevitable water gain. The results speak for themselves, and they seem also to be exactly what should be expected.

The writers, from their own and much other evidence, are in hearty agreement that the strength of the concrete is a poor criterion of its bond resistance and agree that there is a rather pressing need for the selection of a suitable bond specimen. While vertically cast pull-out specimens cannot be expected to give anything like the same bond strength values as will companion horizontally cast beams, there are consistent indications that the values will be proportional and that a simple pull-out type of specimen at some designated length or L/D ratio can be made to give very reliable bond strength indications.

From his early tests Abrams recommended such a specimen with an L/D of 8. The writers feel that there are several items to be considered further before deciding upon the most desirable ratio but that something between 10 and 20 might be satisfactory for the higher yield point steels now generally used.

It is possible that the push-out specimen will prove to be equally as good as the pull-out with the added advantage that it is a simpler one to fabricate and test. A number of the current investigators are working with push-out tests, and additional useful evidence should soon be available. In comparing push-out and pull-out tests, a fundamental difference is that the bar at point of entry is in compression for the push-out specimen, and is in tension for the pull-out. Because of the Poisson ratio effect or tendency of the compression in the bar to swell it against the concrete, instead of drawing down or decreasing the diameter of the bar and pulling it away from the concrete, the push-out specimens should give higher resistances than do pull-outs. Glanville found this to be the case.

The writers' bond tests have been conducted as part of a cooperative investigation between the Highway Research Board and the Iowa Engineering Experiment Station. Messrs. W. M. Dunagan, Glenn Murphy, and G. C. Ernst (now of Maryland) have assisted in various ways. The tests have all been on high elastic limit (rail steel) bars supplied by the Laclede Steel Co. of St. Louis.

SHORT-TIME CREEP TESTS OF CONCRETE IN COMPRESSION

By Russell S. Jensen[1] and Frank E. Richart[1]

Synopsis

The tests reported were made to study the creep that occurs in concrete in compression in relatively short periods of time, varying from 1 to 30 min., under various intensities of stress. The testing apparatus consisted of carbon pile telemeter gages for measuring loads and strains, and an oscillograph recorder.

The tests were made on standard 6 by 12-in. cylinders, with concrete mixtures of four grades of strength. The schedule provided a comparison of the effects of gravel and limestone aggregates, and of hand-tamped and vibrated concrete.

The tests showed no apparent effect on creep due to variations in cement paste ratio, when water-cement ratio was held constant. Vibrated concrete showed slightly greater creep than hand-tamped concrete, and limestone concrete produced much higher creep than gravel concrete. The creep, in general, was roughly proportional to load for stress below one-half the concrete strength; beyond this point the creep increased at an accelerating rate. Comparisons of the test results with published data of long-time tests indicate a continuity between the two and support the view that long- and short-time creeps are fundamentally the same in nature, though the former may be modified by moisture changes and similar conditions which do not exist in the short-time observations.

Object of Tests

The tests reported herein are a continuation of studies of the effect of testing speed on the properties of concrete reported in 1936.[2] One phase of those tests included observations of the plastic deformation or creep of the concrete that took place during very small time intervals. These tests were inspired, in part, by similar observations by Glanville,[3] in England. The 1936 tests showed that measurable creep occurred in test cylinders loaded for 1 min., even at loads only one-eighth of the ultimate strength. The present series was planned to extend and verify these observations, using a larger range of materials and time intervals.

The principal variable studied was the relation between the time of loading and observation and the creep resulting in concretes of different water-cement and cement-paste ratios. One group of tests was also made to study the effect of different concrete aggregates; another group to compare the effects of hand tamping and vibration of the cement.

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[1] Test Assistant, Theoretical and Applied Mechanics, and Research Professor of Engineering Materials, respectively, University of Illinois, Urbana, Ill.

[2] Paul G. Jones and F. E. Richart, "The Effect of Testing Speed on Strength and Elastic Properties of Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 380 (1936).

[3] W. H. Glanville, "Studies in Reinforced Concrete, III. Creep or Flow of Concrete Under Load," *Technical Paper No. 12*, Building Research Station, England (1930).

Acknowledgment.—The project originated in the Society's Committee C-9 on Concrete and Concrete Aggregates, received the sponsorship of the Section on Effect of Speed of Testing of the Society's Committee E-1 on Methods of Testing, and was given further support through a grant of funds by Committee E-9 on Research. The tests were conducted by the Engineering Experiment Station, University of Illinois, in cooperation with the American Society for Testing Materials. The Society was represented by an Advisory Committee consisting of M. O. Withey, P. G. McVetty, and D. E. Parsons. The project was under the administrative direction of M. L. Enger, Director of the Engineering Experiment Station and F. B. Seely, Head of the Department of Theoretical and Applied Mechanics at the university. Acknowledgment is also due to O. S. Peters, Washington, D. C., who furnished the telemeter apparatus used.

OUTLINE OF TESTS

The program of tests involved the testing of a group of standard 6 by 12-in. concrete cylinders, varying in mix from very lean to very rich. All were tested at the age of 28 days. Most of the essential information regarding the materials and the program of tests is given in Table I.

TABLE I.—SCHEDULE OF SHORT-TIME CREEP TESTS.

Three 6 by 12-in. cylinders of a kind for creep tests, additional for strength tests. Cylinders given standard moist curing, 28 days. Strength tests made on companion cylinders to determine ultimate load, then creep tests made by applying load in increments (each 10 per cent of ultimate) for period of 1, 5, or 30 min., as indicated. Loads and strains recorded by oscillograph, using telemeter gages.

Mix	Creep Time Interval, min.	Water-Cement Ratio, by Volume	Proportions by Weight Cement: Fine: Coarse	Aggregate	Paste Ratio	Slump, in.
A 1	1, 5, 30	1.40	1:4 :5	Sand and Gravel	0.270	6.3
A 2	5	1.40	1:3.33:4.17		0.308	7.3
A 3	5	1.40	1:5 :6.25		0.229	0.5
B 1	1, 5, 30	1.10	1:3.25:4		0.279	5.1
B 2	5	1.10	1:2.71:3.33		0.317	6.7
B 3	5	1.10	1:4.06:5		0.239	0.3
C 1	1, 5, 30	0.80	1:2.25:2.75		0.312	5.7
C 2	5	0.80	1:1.88:2.29		0.353	6.3
C 3	5	0.80	1:2.81:3.44		0.267	0.7
D 1	1, 5, 30	0.50	1:1 :1.25		0.435	1.6
D 2	5	0.50	1:0.83:1.04		0.483	6.5
D 3	5	0.50	1:1.25:1.56		0.383	1.2
LA 1	5	1.40	1:4 :5	Limestone	0.273	0.7
LB 1	5	1.10	1:3.25:4		0.282	0.2
LC 1	5	0.80	1:2.25:2.75		0.316	0.2
LD 1	5	0.50	1:1 :1.25		0.440	0.3
VA 3	5	1.40	1:5 :6.25	Sand and Gravel	.....	0.5
VB 3	5	1.10	1:4.06:5		.....	0.3
VC 3	5	0.80	1:2.81:3.44		.....	0.7
VD 3	5	0.50	1:1.25:1.56		.....	1.2

The tests are grouped into three series Series 1 was intended to provide comparisons of the effect of water-cement ratio and quantity of paste in the mixture; with four basic mixes, the cement content was increased or decreased to provide a variation in paste ratio with each value of water-cement ratio. This group of specimens was made with sand and gravel aggregates. A second group was made with limestone aggregates, to study any possible effect on creep. The third group of cylinders was fabricated by use of a small "Viber" internal vibrator, for comparison with the previous lots which were hand tamped into the molds. The paste ratios (ratio of volume of cement paste to total volume of concrete) are given in Table I.

Materials:

Marquette portland cement was used in all cylinders. The sand and gravel were from Wabash river pits. The limestone was from Kankakee, Ill. Fineness moduli for these aggregates are as follows:

	FINE AGGREGATE	COARSE AGGREGATE
Sand, gravel.....	3.10	6.65
Limestone.......	3.92	5.93

The gravel was of $\frac{3}{4}$-in. maximum size, the other, $\frac{1}{2}$ in. All aggregates were proportioned dry. All concrete was machine mixed for 4 min.

Testing Procedure:

Preparatory to the creep tests, companion cylinders were tested to determine the strength and stress-strain relation for each mix. All tests were made in two Olsen testing machines using head speeds (idling) of 0.05 in. per min. or less.

The creep tests were made as described in the paper by Jones and Richart[2] previously mentioned. Loads and strains were measured by means of carbon resistor telemeter gages, and autographic records were taken by means of an oscillograph recorder having super-sensitive elements, which provided a satisfactory precision of measurement. A description of these instruments, their calibration and precision is given in the 1936 paper.[2]

In making the creep tests, after the strength of the concrete had been found from companion cylinders, loads were applied to the test cylinder in increments of 10 per cent of the ultimate strength. Each load increment was held either 1, 5, or 30 min., and during this interval, observations of load and strain were autographically recorded on an oscillograph film. The change in load was negligible, showing a slight decrease at the higher values, but the increase in strain was very apparent and seemed to be consistent and systematic. The change in strain during the period that each increment of load was applied has been scaled from the photographic records. It may be noted here that the precision of strain reading is estimated at 0.000005 in. per inch, while the load readings could be read with a precision of 15 to 20 lb. per sq. in. One feature of these tests should be clearly understood. After creep readings had been taken on a cylinder at 10 per cent of the estimated maximum for the desired period of time, another 10 per cent increment was applied to the same cylinder, and so on until the cylinder failed. The creep measured at the higher loads is probably not the same as it would have been on a cylinder not previously held under load. Another interesting point is that, with the loads held for 5 or 30 min., many cylinders failed at 80 and 90 per cent of the maximum load found for companion cylinders. This may be explained, in part, by natural variations in strength among the group of companion cylinders. However, it is known from other tests that at 80 to 90 per cent of the ultimate, the material is in an advanced stage of plastic deformation and distortion, so that these reduced strengths are not without explanation. In a few cases, too, the strength of the test cylinders exceeded the capacity of the testing machine used in the creep tests, and the test was not carried to the maximum load.

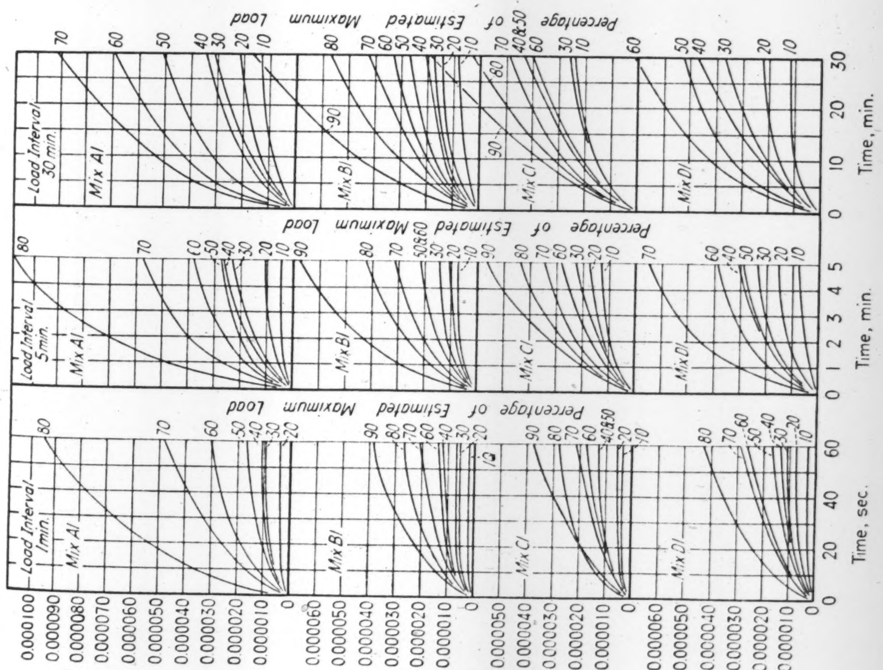

FIG. 1.—Creep-Time Curves for Gravel Concrete, with Three Loading Periods.

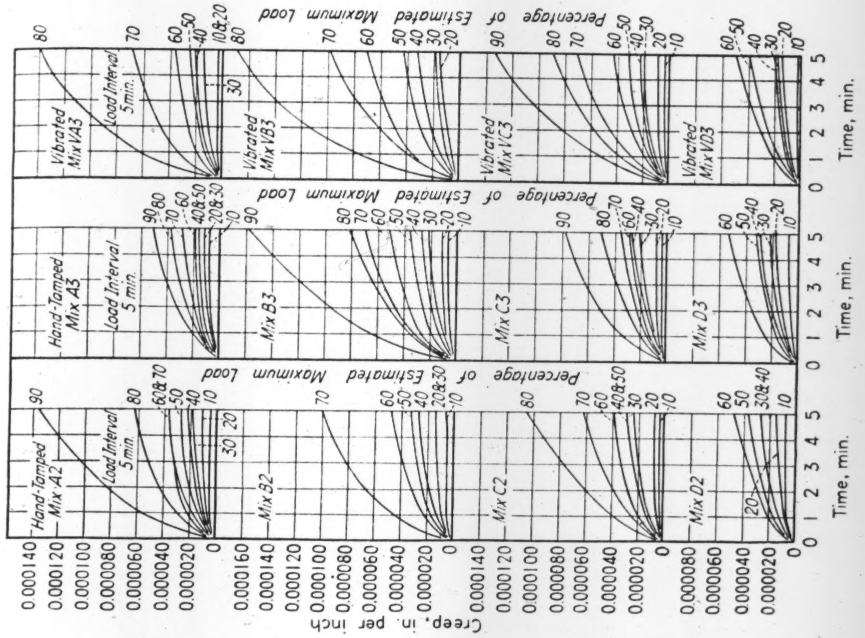

FIG. 2.—Creep-Time Curves for Hand-Tamped and Vibrated Concrete.

RESULTS OF TESTS

Figure 1 presents some of the results of series 1, in which four grades of concrete, and three time intervals, 1, 5 and 30 min., were used. It is evident from a study of these creep curves that the amount of creep increases with the time interval after loading, but the flow is undoubtedly greatly affected by the previous loads that have been applied to the cylinder. Thus, comparing cylinders A 1 at 50 per cent ultimate load, the creep after 1 min. of loading is 0.0000165, 0.0000146 and 0.0000075 for the cylinders loaded at previous load increments for intervals of 1, 5, and 30 min., respectively. This general result is found in all of the curves, although the total amount of creep for the entire period shown increases very definitely for the longer periods of loading.

Comparing the creep found with the various concrete mixtures, it is evident that the creep did not vary in any consistent relation to the strength, at a given percentage of the ultimate load, although there was a decided tendency toward increase in creep with the stronger mixes. The creeps at 80 and 90 per cent of the estimated ultimate load are not particularly reliable, for the reason that the ultimate load used to compute the load increments was determined from companion cylinders; hence if the individual cylinder under observation varies in strength by 10 per cent from the others, the estimated 90 per cent value may actually be anywhere from 80 to 100 per cent. Since it is evident that the creep becomes very great as the cylinder approaches failure, its value at these higher increments is very likely to be erratic.

Figure 2 shows creep-time curves for other mixtures described in Table I, and the middle and right-hand groups of curves, in particular, afford a comparison of hand-tamped and vibrated concrete of identical mixtures. A study of these curves shows that in nearly all cases there was definitely greater creep in the cylinders that were vibrated than in those that were hand tamped. The average excess of creep for vibrated cylinders over that for hand-tamped ones was about 17 per cent at loads of 60 per cent of the ultimate strength, and less. There seems to be no obvious explanation of this result.

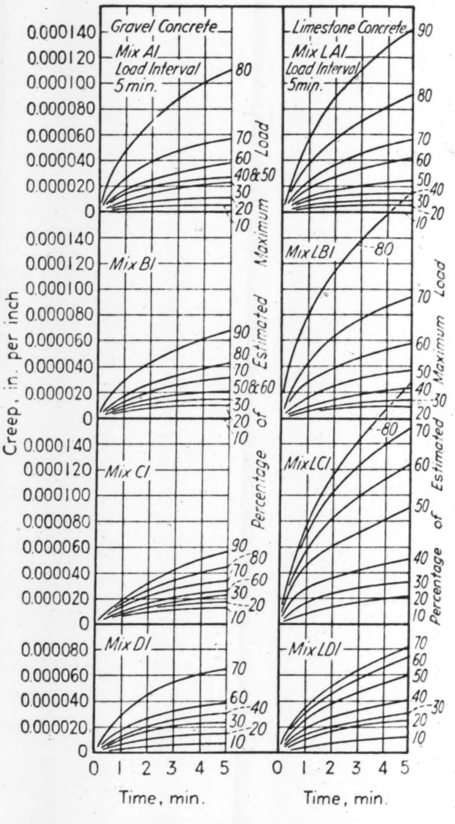

FIG. 3.—Creep-Time Curves for Gravel and Limestone Concrete.

Figure 3 shows curves for cylinders of similar mixtures made with sand-gravel and limestone aggregates. A comparison of these curves shows that for all but the "A" mixes, the cylinders made with limestone aggregates showed by far the most creep. Considering the creep due to loads of 60 per cent ultimate and less, the average excess for limestone concrete over gravel concrete was nearly 60 per cent. There seemed to be no appreciable difference in the shape of the creep-time curves for the two materials.

Creep-Time Relations:

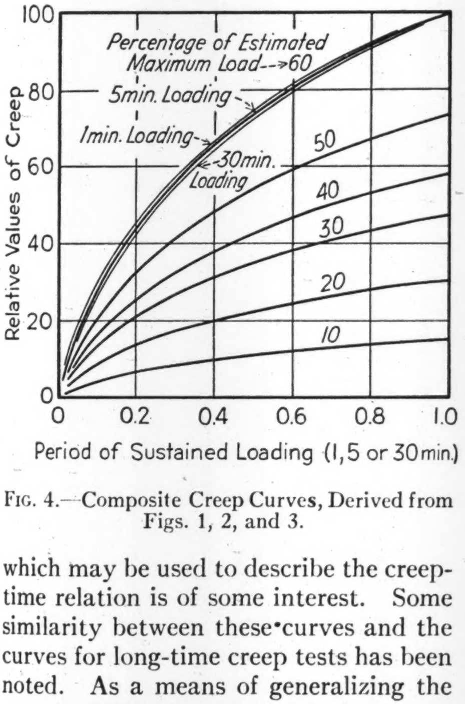

FIG. 4.—Composite Creep Curves, Derived from Figs. 1, 2, and 3.

The shape of the creep-time curve, or in other words the type of equation which may be used to describe the creep-time relation is of some interest. Some similarity between these curves and the curves for long-time creep tests has been noted. As a means of generalizing the curves of Figs. 1 to 3, composite curves have been determined in the following manner: First, the ordinates of each curve, for about six points on the time-axis were determined. Average values of all ordinates, for load increments up to 60 per cent of the ultimate, were found for each of the three time intervals used in the creep tests. Expressing these ordinates as a percentage of the value at the end of the loading period, and expressing all time intervals as a percentage of the loading period, there resulted values of the three composite curves in the upper part of Fig. 4. It will be noted that these three curves are very nearly identical, with the curve for 1-min. loading interval above, the one for the 30-min. interval below, and the one for the 5-min. interval falling on the average of the other two. This average curve is not far from a second-degree parabola with the vertex at the origin of coordinates. The other curves of Fig. 4 are based upon the upper average curve, and were derived by determining the average creep interval in Figs. 1 to 3, produced by increments of loading up to 60 per cent of the ultimate. Taking the 60 per cent increment as a basis of comparison, the other curves were drawn with all ordinates to the proportionate scale thus determined. It may be noted that while the creep values above the 60 per cent load are erratic and in many cases not determined, the average of those found is as follows, taking the value for 60 per cent load as 1.0:

LOAD INCREMENT, PER CENT OF ULTIMATE	RELATIVE VALUE OF CREEP
60	1.00
70	1.38
80	2.05
90	3.43

Obviously, while the creep was nearly proportional to the load up to 50 per cent of the ultimate, the departure from proportionality increased at a rapid rate beyond this point.

It is of interest that the long-time relations between creep and time of loading, as summarized by Shank,[4] have been reported by him to follow a third-degree parabola with a fair degree of accuracy. Shank's equation, for concrete of ordinary proportions, previously moist cured for 28 days and unstressed up to the time of testing, is as follows:

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[4] J. R. Shank, "The Mechanics of Plastic Flow of Concrete," *Proceedings*, Am. Concrete Inst., Vol. XXXII, p. 149 (1936).

$$y = 0.13 \sqrt[3]{x} \dots\dots\dots (1)$$

where y = the creep for a stress of 1 lb. per sq. in., and

x = the time, in days, after load is applied.

Most of the observations upon which this equation was based extended from a week to a year or more; some of the data fell between second- and third-degree parabolas.

As noted before, the creep of the present test pieces was obviously affected by the previous increments of sustained loading and creep. It would be expected to be considerably less than the corresponding creep for a cylinder that had not been previously loaded. A few computations have been made to see whether Eq. 1 will give values that agree even roughly with the test results.

Table II presents values of creep, summarized from the tests on hand-tamped gravel concrete. The table also presents computed values of the creep per 1 lb. per sq. in. of stress, assuming proportionality to exist between creep and intensity of loading. Considering that mixes A and B represent "ordinary" concrete, it is seen that there is fair agreement between the average test values of y and the values found from Eq. 1 as shown by the last two columns of the table.

TABLE II.—SUMMARY OF CREEP VALUES FROM TESTS.

Values given are for a specific load increment, not the cumulative creep for all loads up to the indicated increment.

Mix	Concrete Strength, lb. per sq. in.	Loading Period, min.	Creep Due to Applied Stress, millionths in. per inch						Average	Value y from Shank's Equation[a]
			Percentage of Concrete Strength							
			10	20	30	40	50	60		
MEASURED VALUES										
A 1, 2, 3	1975	5	6.0	10.8	18.0	21.4	22.6	31.4		
B 1, 2, 3	3035	5	6.7	12.0	17.0	26.0	29.9	40.2		
C 1, 2, 3	4965	5	6.4	10.8	22.4	27.4	25.0	31.4		
D 1, 2, 3	8065	5	7.4	16.0	23.5	28.5	32.8	46.4		
A 1	2100	1	0	3.7	9.3	10.5	16.5	30.1		
B 1	3110	1	2.1	5.5	6.6	12.2	12.8	13.5		
C 1	4810	1	3.4	5.5	12.7	11.3	13.0	17.5		
D 1	8170	1	2.9	7.3	10.3	13.2	17.0	26.0		
A 1	2100	30	13.7	21.3	31.5	34.2	51.1	69.4		
B 1	3110	30	8.8	14.5	17.8	21.0	27.0	33.0		
C 1	4810	30	24.0	26.0	41.3	43.0	34.3	40.3		
D 1	8170	30	12.0	22.5	37.0	47.0	52.0	71.0		
CREEP DUE TO STRESS OF 1 LB. PER SQ. IN. (COMPUTED)										
A 1, 2, 3	1975	5	0.031	0.027	0.031	0.027	0.023	0.027	0.028	0.020
B 1, 2, 3	3035	5	0.022	0.020	0.019	0.021	0.020	0.022	0.021	
C 1, 2, 3	4965	5	0.013	0.011	0.015	0.014	0.010	0.010	0.012	
D 1, 2, 3	8065	5	0.009	0.010	0.009	0.009	0.008	0.010	0.009	
A 1	2100	1	....	0.009	0.015	0.012	0.016	0.024	0.013	0.012
B 1	3110	1	0.007	0.009	0.007	0.010	0.008	0.007	0.008	
C 1	4810	1	0.007	0.006	0.009	0.006	0.005	0.006	0.007	
D 1	8170	1	0.004	0.004	0.004	0.004	0.004	0.005	0.004	
A 1	2100	30	0.065	0.051	0.050	0.041	0.048	0.055	0.052	0.049
B 1	3110	30	0.028	0.023	0.019	0.017	0.017	0.018	0.021	
C 1	4810	30	0.050	0.027	0.029	0.022	0.014	0.014	0.026	
D 1	8170	30	0.015	0.014	0.015	0.014	0.013	0.014	0.014	

[a] These values computed from Shank's equation, $y = 0.13 \sqrt[3]{x}$, for *ordinary* concrete, x = days of sustained loading

It seems reasonable, from the general shape of the curves and from the degree of accordance found between these tests and the others represented by Eq. 1, to conclude that the same phenomenon of inelastic deformation under applied load applies to all. Undoubtedly long-time creep is affected by moisture conditions, which modify the results, but the fundamental cause of the creep would seem to be the same in both cases.

The data of Table II present another important relation between creep and the strength of the concrete. While there is no great variation in creep with concrete strength, at loads representing equal proportions of the ultimate strength, when the creep is expressed in terms of a load of unity, it is very obvious that this specific creep decreases rapidly as the strength increases. Thus comparing mixes A and D, in which the strength is about in the ratio 1 to 4, the specific creep of the stronger specimens is about one-third that of the weaker ones. This seems logical and is supported by tests of long-time creep available in the literature on the subject, although this relation is not provided for in Eq. 1, beyond the limitation that it applies only to "ordinary" concretes.

The data of the foregoing tests suggest the desirability of further studies in which creep observations might be taken on test pieces within a few seconds after load is applied and the observations then continued for a year or more to tie together the two creep phenomena, which have been believed by some to be of entirely different nature.

SUMMARY

The results of the tests may be summarized briefly, as follows:

1. The tests varied from the usual creep test in that loads were applied in increments, with creep measurements under the sustained load at each increment. The increments were 10 per cent of the estimated maximum load. The creeps thus measured were evidently affected by the plastic deformation of the cylinder under previous load increments.

2. The telemeter gages used for measuring strains and loads seemed to give satisfactory results.

3. There seemed to be no effect on creep produced by varying the cement-paste ratio, when the water-cement ratio was held constant.

4. A variation in water-cement ratio, and consequently of strength, produced a pronounced effect on the creep per unit of load, which may be denoted as specific creep. The specific creep varied in nearly inverse proportion to the strength.

5. Vibrated concrete, as used in these tests, exhibited slightly larger amounts of creep than corresponding hand-tamped concrete of identical materials and proportions.

6. The creep found for similar concretes made with limestone and sand-gravel aggregates averages nearly 60 per cent greater for the former than for the latter.

7. The creep at successive increments of load varied, on the average, approximately in proportion to the intensity of applied load, up to about 50 per cent of the ultimate strength of the concrete; beyond this point the creep increased at a rapidly accelerating rate.

8. The average load-creep curve from these tests is approximately a second-degree parabola, thus varying from the cubic parabola generally accepted as representing the long-time creep-time relation. However, a comparison of the average test results with results computed from the cubic equation shows a reasonably good agreement between the two, and gives support to the idea that the creep observed in the short periods of time used in these tests is of the same general character as the long-time creep or time yield generally observed over periods measured in months and years.

DISCUSSION

MR. CHARLES E. WUERPEL.[1]—May I ask whether the authors assume that the final results for a 30-min. period of loading might be added to other results obtained from a 30-min. initial reading to obtain the total creep in six months' time?

We ran a series of tests on plastic flow in which we started observations of creep 30 min. after the application of the load; I want to know whether it is conceivable that the initial creep should be added to show the total creep in a year.

MR. F. E. RICHART.[2]—It seems reasonable that the creep for the initial 30 min. of loading might be used as an additive correction to creep test data obtained from observations started 30 min. after application of load. Our only information applying directly to this condition is that for the 10 per cent maximum loads in Fig. 1. The creep due to a 5-min. application of load is nearly the same in the center and right-hand group of curves, at 10 per cent load. Of course, at other load increments, the same agreement can hardly be expected, because the treatment has been different at the previous load increments and this undoubtedly decreases the creep in those specimens that have been held under previous sustained load increments the longer periods of time. However, for test specimens that had not been previously loaded, I believe the two amounts of creep might be added directly, as Mr. Wuerpel suggests.

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DRYING SHRINKAGE OF CONCRETE AS AFFECTED BY MANY FACTORS

By Roy W. Carlson[1]

Synopsis

This paper is a progress report on a long-range program of research aimed toward a better understanding of concrete shrinkage.

Among the results presented, it is shown that for a common glacial gravel and a standard cement, neither cement content, nor gradation of aggregate, nor duration of preliminary moist curing has much effect on shrinkage.

It is indicated that invisible cracking of mortar between aggregate particles is common in concrete subjected to drying. Differences in extent of cracking often may be the distinguishing feature between concretes of high and low shrinkage. To test this hypothesis, attempts were made to reduce shrinkage by reducing the tensile strength of the mortar in concrete. Small amounts of ground mica were added to concrete mixes. Substantial reductions in shrinkage were obtained.

Type of aggregate is shown to have a large effect on concrete shrinkage. Among the pure-mineral aggregates, dense grades of quartz, feldspar, dolomite, and limestone are outstanding in producing low shrinkage. Hornblende and pyroxene, which are ferromagnesium silicates, produce concrete of high shrinkage. Among the aggregates of mixed composition, slate and sandstone produce concrete of very high shrinkage. Granite falls intermediate between aggregates producing high and low shrinkage in accordance with its varied constituents. From incomplete studies to date, it appears that the rigidity, or modulus of elasticity, of the aggregate largely determines whether or not it will produce concrete of high or low shrinkage.

Since 1935, when a paper on "The Chemistry and Physics of Concrete Shrinkage"[2] was presented, many tests have been made to investigate a wide variety of factors that were reasoned to have an effect on shrinkage. Although more than 1000 additional specimens have been tested, the work is far from complete. The almost limitless combinations of influencing factors magnifies both the scope of the work and the possibility of useful results.

The present paper offers results of certain selected tests that may have a bearing on the ultimate control of drying shrinkage. The effects on concrete shrinkage of many factors are discussed, partly for what practical value they may have, and partly for enlisting the criticism of those interested in concrete shrinkage. This paper can only be considered as a progress report. Modifications of plans for research on shrinkage are indicated for future work. Some lines of attack are discouraged by results to date, while others seem to offer much promise.

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[1] Associate Professor of Civil Engineering, Massachusetts Institute of Technology, Cambridge, Mass.

[2] R. W. Carlson, "The Chemistry and Physics of Concrete Shrinkage," *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part II, p. 370 (1935).

METHODS OF TEST

The shrinkage tests reported herein are confined to measurements of the contraction that occurs when specimens of concrete are stored in air of 50 per cent relative humidity and 74 F. The specimens are kept moist for a prescribed period of time after mixing to simulate job requirements for curing. Thereafter, they are stored in a room maintained at constant temperature and humidity where they lose moisture and contract. Stainless steel plugs are cast into the ends of specimens to permit easy measurement of contraction. An extensometer is used in which the indicating device is a dial graduated in ten-thousandth inches. The effect of a change in length of the extensometer itself is eliminated by taking simultaneous readings on a steel bar of fixed length.

The specimens, except where noted, are 3 by 6-in. cylinders having a net gage length of 5 in. between the end plugs. In a few cases, 1½ by 1½ by 12-in. bars are used either for the purpose of obtaining quick drying or of reducing the tendency toward surface cracking. In the earlier tests, specimens were in duplicate for each condition, but in later tests, triplicate specimens are being made.

The accuracy of readings is in the neighborhood of 10 millionths of the length of a 6-in. cylinder. Duplicate cylinders often differ by more than this amount, especially when internal cracking of concrete is indicated. The element of chance that governs the cracking probably prevents any two specimens from cracking equally. When internal cracking is not indicated, duplicate specimens show almost equal shrinkages.

Shrinkages are reported in millionths of the length. In the case of a 5-in. gage length, the least reading that can be estimated on the extensometer dial amounts to 2 millionths. The least difference in shrinkage measurement that has any significance is 10 millionths, which amounts to a little more than 0.01 in. per 100 ft. of length. In many cases where cracking of specimens may be involved, results cannot be duplicated within about 50 millionths or more.

The aggregates for nearly all specimens were sieved into all of the standard sieve sizes and recombined as desired. Most gradations were of the "ideal" type, in which there was a systematic variation in the amount of successively smaller sizes.

SHRINKAGE OF NEAT CEMENTS

General Behavior of Neat Cements:

After cement has hydrated, it consists partially of crystalline centers that are believed to be immune to the effect of ordinary drying. The crystalline centers are made up mainly of the unhydrated remainders of cement grains and of calcium hydroxide crystals that are a by-product of hydration. The remainder of the hydrated cement is gelatinous material, which is finely porous and susceptible of large volume changes upon wetting and drying. If the water-cement ratio is in the range encountered in average concrete, the gelatinous material has a greater volume than the crystalline material.

The shrinkage in length of the pure gel of portland cement due to drying in air is estimated at well over 1 per cent under average conditions. If the water-cement ratio is very high, the gel will be of coarse texture and such gel may have a drying shrinkage of several per cent, although the shrinkage of pure cement gel has not been measured up to the present time.

The true shrinkage of neat cement is difficult to measure because cracking or checking of specimens may occur without being detected. The results for neat cements discussed herein were obtained mainly on bars $1\frac{1}{2}$ by $1\frac{1}{2}$ by 12 in. in size and in a few cases very fine cracks were barely visible. In other cases, cracking was suspected but not visible. The effect of cracking is to permit shrinkage to occur without fully reducing the over-all length of a specimen; therefore reported shrinkages will be too low.

Typical Shrinkage of Neat Cements:

In the following table are typical measured shrinkages of a variety of neat cements, all having a water-cement ratio of 0.30 by weight. Note that the high-early-strength cement exhibits less shrinkage than does the standard cement. Also, the effect of air separating the majority of particles finer than 5 μ from the high-early-strength cement is to increase the shrinkage. The high shrinkage of low-heat and portland-puzzolan cements is characteristic of these types of cement which are believed to produce large percentages of gel. Cracking of some of the specimens represented in the table is suspected, but there is no adequate proof at this point; no cracks were visible:

SHRINKAGE OF NEAT CEMENTS IN AIR AT AGE OF 1 YR.

CEMENT	SHRINKAGE, MILLIONTHS
Standard, 7 per cent C_3A, 1400 sq. cm. per g.	2150
High-early-strength cement, containing 29 per cent minus 5 μ	2335
Air separated to 5 per cent minus 5 μ	2670
Low heat, 5 per cent C_3A, 1900 sq. cm. per g.	2870
Portland puzzolan	3150

Effect of Duration of Moist Curing:

A complicating factor in all shrinkage studies is the variable effect of duration of preliminary moist curing. It would be natural to expect a long period of moist curing to harden a cement mixture and thus to reduce the magnitude of its desired shrinkage. But the continued hardening strengthens the cement gel and thus permits it to attain a larger percentage of its desired shrinkage against the restraining influence of the inert grains. It is therefore conceivable that prolonged moist curing may decrease shrinkage to a different degree depending on the relative importance of the two opposing effects. In concretes, particularly those in which cracking between aggregate particles is prevalent, prolonged moist curing may increase subsequent (measured) shrinkage by reducing the cracking, while in neat cements there is usually more or less reduction in shrinkage with prolonged moist curing. The following table shows the shrinkages of selected neat cements to illustrate the variable effect of prolonged moist curing:

EFFECT OF CURING PERIOD ON SHRINKAGE OF NEAT CEMENTS.

Cement	Water-Cement Ratio, by weight	3-month Shrinkage, millionths	
		After 2 days Moist Curing	After 28 days Moist Curing
A (standard)	0.40	3500	2240
B (75 per cent of cement A, 25 per cent of dolomite fines)	0.40	3120	2580
C (50 per cent of cement A, 50 per cent of dolomite fines)	0.40	2860	2880
D (modified, 6 per cent C_3A)	0.30	1490	1380
D (modified, 6 per cent C_3A)	0.60	2220	2200

The more common effect of prolonged moist curing reducing the shrinkage is exhibited by cement A, which is a representative standard cement. When this cement was adulterated with 25 per cent of inert fines, the effect of prolonged moist curing was lessened. When the adulteration was extended to an equal amount of inert fines and cement, prolonged moist curing did not reduce the shrinkage at all.

Results on a modified cement (cement D) are included in the above table to avoid the conclusion that the effect of moist curing can only be altered by adulterating a cement. In this case, little or no reduction in shrinkage is obtained from longer moist curing, even for the straight cement.

Effect of Water-Cement Ratio:

Water plays a most important part in the shrinkage of cement mixtures. A knowledge of the reason for water having such a large effect contributes to the understanding of the shrinkage of concrete. When cement hydrates, it fills the space originally occupied by the mixing water with hydration products, mainly gel. The volume of hydration products formed is therefore the volume of the water plus the volume of cement that is consumed by the hydration. The simplest picture of the effect of changing water content is to assume cement to hydrate entirely. Then a definite volume of non-shrinking hydration products is formed from each pound of cement and the remainder of the volume of the hydrated paste is gelatinous material. Now, when the water content is raised, the amount of non-shrinking material remains the same and the volume of gelatinous material must be increased by the full volume of the added water. The concept of the gelatinous constituent adjusting its volume to fill all available space (excepting the relatively large air voids) is fundamental. Thus, for each 1 per cent increase in water content, there is a 1 per cent increase in volume of gelatinous material per unit volume of paste. Furthermore, the greater quantity of gel is necessarily of coarser texture and of higher shrinkage tendency. Although the cement does not generally fulfill the assumption of hydrating completely, the difference due to incomplete hydration will not often be large.

It would be desirable to know the shrinkage of neat cements for the relatively high water-cement ratios that prevail in concrete. It is possible to make specimens of wet mixtures of neat cement by stirring occasionally to prevent sedimentation before hardening. In the higher water-cement ratios, however, cracking is common during drying, and measurements may therefore be misleading.

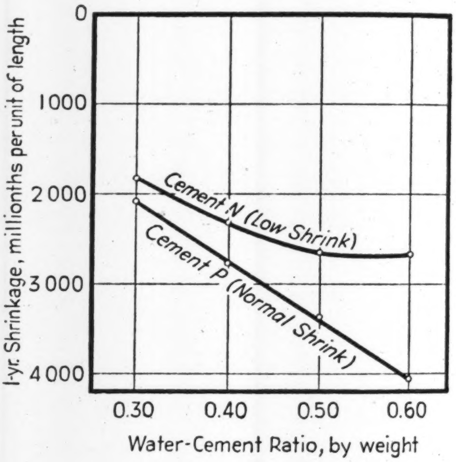

FIG. 1.—Shrinkage of Neat Cement.

In Fig. 1 are shown the measured shrinkages on neat specimens of two cements with varying water contents. The results suggest that for cement N there may have been cracking of specimens that increased as the water content was raised; no increase in shrinkage was found for a water-cement ratio of 0.60 over that of 0.50. This cement produces concrete of low measured shrinkage, which is included among data to be presented later. Cement P, on the other hand, exhibits a shrinkage that increases with increasing water-cement ratio in a logical manner. This cement produces concrete of relatively high shrinkage.

Effect of Individual Compounds of Cement:

Neat specimens were made of the pure compounds—tricalcium silicate (C_3S), dicalcium silicate (C_2S) and tricalcium aluminate (C_3A)—each ground to about the fineness of cement and used as cement. The results were as follows:

C_3S expanded slightly under moist curing, then contracted in air to about 70 per cent of the average shrinkage of portland cement.

C_2S remained constant in length under moist curing, then contracted in air at about twice the rate of average cement, but the specimens soon cracked and and reliable measurements of ultimate shrinkage could not be obtained.

C_3A expanded greatly under moist curing, then contracted rapidly in air, the specimens cracking to such an extent that later measurements had no meaning.

Of the three compounds tested, C_3S exhibited the greatest volume constancy and C_3A gave the poorest performance. These results are in agreement with those of Woods[3] and of Bogue,[4] who determined the relative effect of these compounds in cement.

SHRINKAGE OF CONCRETE

Difference Between Neat Cement and Concrete:

Concrete is neat cement that has been diluted with aggregate. If it were possible to dissolve the aggregate from a concrete and leave a skeleton of neat cement, that skeleton would tend to shrink as much in over-all dimensions as would a corresponding solid specimen of neat cement. Likewise, if the aggregate were readily compressible, the concrete would shrink as much as neat cement. This fact was demonstrated by making a concrete in which the aggregate consisted of porous but non-absorbent rubber particles. This concrete and the corresponding neat cement contracted in air almost equally, and about 8 times as much as did ordinary concrete.

When neat cement is diluted with aggregate that is not readily compressible, as in ordinary concrete, the drying shrinkage of the concrete is but a fractional part of that of the neat cement. Evidently the aggregate particles reinforce the skeleton against contracting. The ability of the aggregate particles to restrain the cement paste and resist shrinkage depends upon several important factors:

1. The relative extensibility of paste and compressibility of aggregate,
2. The bond or contact between paste and aggregate,
3. The plasticity of the paste (may be included with 1),
4. The degree of cracking of the cement paste, and
5. Volume change of aggregate particles due to drying.

Some simple test results will show the large effect of the aggregate and also will demonstrate the fact that all aggregates are not equally effective. In the following table, the shrinkage of neat cement is compared with the corresponding shrinkages of specimens in which the cement is diluted with a single sieve size of siliceous gravel and broken limestone, respectively. All aggregate passed a $\frac{3}{8}$-in. sieve and was retained on a No. 4 sieve.

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[3] H. Woods, H. R. Starke and H. H. Steinour, "Effect of Composition of Portland Cement on Length and Weight Changes of Mortar," *Rock Products*, Vol. 36, June 25, 1933, p. 42.

[4] R. H. Bogue, W. Lerch and W. C. Taylor, "Influence of Composition on Volume Constancy and Salt Resistance of Portland Cement Pastes," *Industrial and Engineering Chemistry*, Vol. 26, p. 1049 (1934).

EFFECT ON SHRINKAGE OF DILUTING CEMENT PASTE WITH AGGREGATE.

Aggregate	Parts Cement	Parts Aggregate	Parts Water	Water, per cent by volume	Paste, per cent by volume	2-yr. Shrinkage in Air, millionths
None (neat paste)	1	none	0.40	56	100	2705
Mixed gravel	1	2.5	0.40	24	44	725
Crushed limestone	1	2.6	0.40	24	44	417

Volume relationships of water, cement, and aggregate in this case are insufficient to explain the difference in shrinkage between the paste and concrete. The reduction in shrinkage due to the aggregate is out of proportion to its relative volume, even granting that the aggregate may be more rigid than the cement paste. Cracking between aggregate particles seems to be a factor.

A clearer picture of the effect of aggregate can be obtained by proceeding step by step from cases where cracking is not indicated to cases where cracking is fairly certain to exist. Such a series is presented in the following table, giving results based on specimens of neat cement diluted with varying sizes of dolomite aggregate. The water-cement ratio was 0.40 throughout, and the mix was 1:1 by weight for all except the control specimens, which contained no aggregate.

EFFECT OF DILUTING CEMENT PASTE WITH DOLOMITE AGGREGATE OF VARIOUS SIZES.

AGGREGATE SIZE	1-YR. SHRINKAGE, MILLIONTHS
None (neat paste)	2710
No. 48 to No. 28 sieves	1190
No. 28 to No. 14 sieves	1240
No. 14 to No. 8 sieves	1220
No. 8 to No. 4 sieves	1160
No. 4 to $\frac{3}{8}$-in. sieves	940
$\frac{3}{8}$-in. to $\frac{3}{4}$-in. sieves	690

Note that for all of the smaller sizes of aggregate the shrinkage is quite uniform, suggesting that in this range the size has no effect on shrinkage. One can assume that in this range of sizes there is no internal cracking and that the shrinkage represents a condition of simple force equilibrium, the paste being in tension and the aggregate being in compression. A simple computation under the assumptions of no cracking and of close contact between paste and aggregate will show the measured results to require that the modulus of elasticity of the aggregate be about three times that of the paste. In view of the effect of plasticity of the paste tending to reduce its "sustained modulus of elasticity," the 3 to 1 ratio appears to be reasonable. The term "sustained modulus of elasticity" is used to denote the ratio of sustained load to elastic plus plastic deformation. In future tests, quantitative data are to be obtained in an attempt to check this deduction.

The reduction in shrinkage caused by the larger sizes of aggregate indicates that, for these materials and conditions, cracking begins when the size of aggregate exceeds about $\frac{1}{4}$ in. It should be stated that these tests were made with a particular cement and aggregate, each of which encouraged cracking.

The possibility of internal cracking of paste between aggregate particles seems to be such an important factor in concrete shrinkage that the foregoing introduction was purposely made before presenting the varied results to follow. It may simplify the understanding of some of the seemingly contradictory results if the possibility of internal cracking is always admitted.

Effect of Cement and Water Contents in Concrete:

In concrete, as in neat cement, the water content is probably the most important single factor affecting shrinkage. Any condition which will permit the use of less mixing water will reduce inherent shrinkage greatly. True, other factors may have a larger effect on the over-all measured shrinkage, but if internal cracks are included as shrinkage, water content will probably be found to be the largest single factor.

For each 1 per cent increase in the quantity of mixing water of a concrete, the shrinkage is increased about 2 per cent. This "double effect" is revealed by a study of a great many tests in which water content was varied. The explanation seems to be that for each 1 per cent increase in mixing water, the volume of gel to be formed in the concrete is increased almost 1 per cent at the same time as the shrinkage tendency of the gel is increased about 1 per cent by the extra water.

Cement content, on the other hand, has a relatively small effect on shrinkage, at least in the case of common gravel aggregates. In general, extra cement increases shrinkage, but the effect varies considerably with the type of aggregate. For the gravel aggregate commonly used in the vicinity of Boston, termed "mixed gravel" herein, cement content was found to have practically no effect on shrinkage when both water content per unit volume of concrete and maximum size of aggregate were maintained constant. For aggregates such as crushed granite and dolomite, which are noted for producing concrete of low shrinkage, larger shrinkage was observed for higher cement content. Figure 2 shows some comparisons of the effect of cement content for a common mixed gravel and for crushed granite. Three brands of cement and three durations of preliminary moist curing are included. It will be noted that concrete containing a high content of low-heat cement and granite aggregate (28-day cure) shows a lower shrinkage than corresponding concrete of lower cement content. Cracking is believed to be a factor, although other possible explanations are not excluded from future studies.

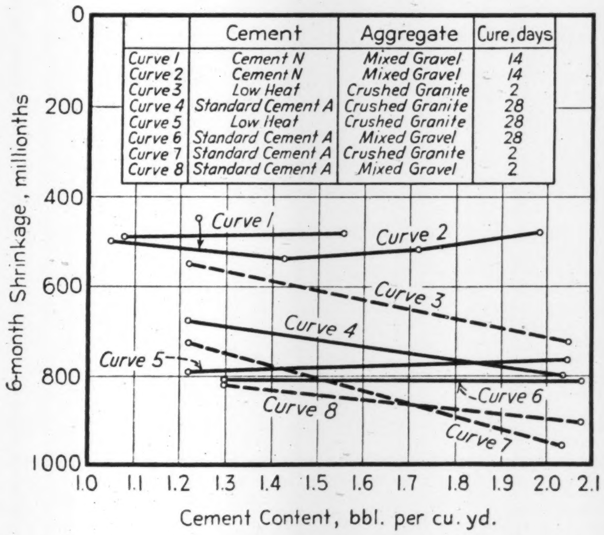

FIG. 2.—Effect of Cement Content on Concrete Shrinkage.

The large importance of water content as compared with cement content (especially in the case of common gravels) was demonstrated forcibly by Vidal and Ehrenburg of the Denver laboratories of the Bureau of Reclamation. They drew lines through points representing concretes of equal shrinkage on a chart in which the ordinates were water contents and the abscissae were cement contents. The lines were nearly horizontal, indicating that as long as the water content per cubic yard was not varied, the shrinkage would be nearly constant for a wide range of cement contents.

Effect of Gradation of Aggregate:

Only a small variation in shrinkage was observed for a wide variety of acceptable gradations of aggregate. The tests involved hundreds of specimens in which systematic as well as extraordinary variations in gradation were introduced while the maximum size of aggregate was maintained at $\frac{3}{4}$ in. and the water-cement ratio was varied only enough to keep the slump constant at 3 in.

TABLE I.—EFFECT OF AGGREGATE GRADATION ON SHRINKAGE OF CONCRETE OF FIXED CEMENT CONTENT.

Gradation	Sieve Analysis, per cent retained on								Water-Cement Ratio, by Weight	Fineness Modulus	6-month Shrinkage, millionths
	3/8-in. Sieve	Sieve No. 4	Sieve No. 8	Sieve No. 14	Sieve No. 28	Sieve No. 48	Sieve No. 100	Pan			
CONTINUOUS GRADATIONS											
No. 1	40.7	24.4	14.6	8.8	5.3	3.2	1.9	1.1	0.62	5.6	650
No. 2	36.6	23.8	15.1	9.8	6.3	4.1	2.6	1.7	0.64	5.4	680
No. 3	31.6	22.1	15.6	11.0	7.8	5.4	3.8	2.7	0.66	5.1	710
No. 4	26.4	20.3	15.6	11.9	9.2	7.0	5.5	4.1	0.69	4.8	740
No. 5	21.2	17.9	15.0	12.6	10.6	8.9	7.5	6.3	0.74	4.4	840
GAP GRADATIONS											
No. 6[a]	29.7	21.6	15.6	11.4	8.2	6.0	4.3	3.2	0.65	5.0	590
No. 7	34.4	22.4	14.7	9.5	0	0	11.6	7.4	0.65	5.0	690
No. 8	37.2	24.2	15.7	0	0	0	14.0	8.9	0.65	5.0	620
No. 9	33.2	21.6	14.1	9.1	0	10.6	6.9	4.5	0.65	5.0	630
No. 10	35.7	23.2	15.1	0	0	12.6	8.2	5.2	0.65	5.0	620
No. 11	41.1	28.8	0	0	0	13.7	9.7	6.7	0.65	5.0	560
No. 12	64.8	0	0	0	0	16.0	11.2	8.0	0.65	5.0	590
GRADATIONS WITH PARTICLE INTERFERENCE											
No. 13[a]	26.4	20.3	15.6	11.9	9.2	7.0	5.5	4.1	0.69	4.8	630
No. 14	28.6	20.0	14.1	9.9	7.0	14.6	3.4	2.4	0.72	4.8	620
No. 15	27.3	19.2	13.5	9.5	20.3	4.6	3.3	2.3	0.73	4.8	650
No. 16	24.1	16.9	35.6	8.4	5.9	4.1	2.9	2.1	0.71	5.1	790
GRADATIONS WITH FINES OMITTED											
No. 17[a]	21.2	17.9	15.0	12.6	10.6	8.9	7.5	6.3	0.74	4.4	740
No. 18	22.6	19.1	16.0	13.5	11.3	9.5	8.0	0	0.72	4.7	730
No. 19	24.6	20.8	17.4	14.6	12.3	10.3	0	0	0.72	5.0	720

[a] Continuous gradation for comparison.

Table I contains measured shrinkages on selected concretes in which the gradation was varied while the cement content was kept constant. The first five are so-called "ideal" gradations. In these gradations, the percentage of each successively smaller size in terms of the next larger size is fixed. Thus in gradation No. 1, the percentage is 60 and in No. 5 it is 84. Gradation No. 1 is coarser than would ever be used in practice unless the cement content were higher than that used. On the other hand, No. 5 is a finer gradation than should ever be used in practice unless the cement content were lower. An appreciable variation in shrinkage appears among these five gradations in accordance with their different water contents. In the range of fineness moduli that would be acceptable in practice, the variation is not large.

The gap gradations included in Table I cover about as large a range as is practicable for the condition of maintaining the same fineness modulus. It may be noted that the percentage of the largest size was varied in the gap gradations from 33 to 65 and the percentage of the finest size was varied from 4.5 to 9 per cent. Due to the fact that the fineness modulus was held constant in this series, the water-cement ratio was also held constant and a slight variation in slump was permitted. In spite of the rather large changes in gradation, variations in shrinkage seldom exceeded the experimental error. Many other gap gradations were tested besides those shown, but with no positive results.

TABLE II.—EFFECT OF GRADATION OF AGGREGATE ON SHRINKAGE OF CONCRETE OF VARIABLE CEMENT CONTENT.

Gradation	Sieve Analysis, per cent retained on								Mix by weight	Water-Cement Ratio, by Weight	Water Volume, per cent[a]	Fineness Modulus	6-month Shrinkage, millionths
	3/8-in. Sieve	Sieve No. 4	Sieve No. 8	Sieve No. 14	Sieve No. 28	Sieve No. 48	Sieve No. 100	Pan					
VARYING MINIMUM SIZE													
No. 1	36.1	23.4	15.4	9.9	6.5	4.2	2.7	1.8	1: 8.5	0.77	18	5.4	500
No. 2	36.8	23.8	15.6	10.1	6.6	4.3	2.8	0	1: 7.5	0.64	16	5.5	440
No. 3	37.8	24.6	16.0	10.4	6.8	4.4	0	0	1: 6.2	0.52	17	5.6	520
No. 4	39.5	25.7	16.7	10.9	7.2	0	0	0	1: 5.0	0.44	17	5.8	520
No. 5	42.5	27.7	18.1	11.7	0	0	0	0	1: 4.2	0.38	17	6.0	480
No. 6	48.4	31.3	20.3	0	0	0	0	0	1: 3.5	0.37	19	6.3	450
No. 7	60.6	39.4	0	0	0	0	0	0	1: 3.0	0.36	20	6.6	430
No. 8	100.0	0	0	0	0	0	0	0	1: 2.5	0.36	20	7.0	390
GAP GRADATIONS													
No. 9[b]	29.7	21.6	15.6	11.4	8.2	6.0	4.3	3.2	1: 6.7	0.65	19	5.0	590
No. 10	34.4	22.4	14.7	9.5	0	0	11.6	7.4	1:12.7	1.15	19	5.0	450
No. 11	38.7	25.2	16.5	10.6	0	0	5.5	3.5	1: 7.6	0.72	19	5.5	550
No. 12	37.2	24.2	15.7	0	0	0	14.0	8.9	1:12.7	1.20	19	5.0	490
No. 13	41.6	27.0	17.5	0	0	0	8.5	5.4	1: 7.6	0.67	18	5.5	540
No. 14	35.7	23.2	15.1	0	0	12.6	8.2	5.2	1:12.7	1.24	20	5.0	450
No. 15	40.6	26.4	17.0	0	0	7.7	5.0	3.3	1: 7.7	0.69	18	5.5	600
No. 16	46.0	0	27.6	0	16.5	0	9.9	0	1:10.0	1.00	20	5.1	490
No. 17	53.3	0	26.6	0	13.4	0	6.7	0	1: 7.7	0.71	18	5.5	600
No. 18	51.0	0	0	30.6	0	0	18.4	0	1:10.0	0.94	19	5.0	620
No. 19	57.1	0	0	28.6	0	0	14.3	0	1: 9.1	0.87	19	5.3	560

[a] Water volume is per unit volume of concrete.
[b] Continuous gradation for comparison.

The effect of particle interference is shown in the third series of gradations listed. In these gradations, there was a large excess of a single size of aggregate, except in the case of the control specimens. It may be noted that the particle interference made higher water-cement ratios necessary to maintain the 3-in. slump. The shrinkage was not materially affected, however, except when the excess particles were in the No. 8 to No. 4 size, in which case the shrinkage was increased. This may have been due to the high air-void content in this mix; its weight per cubic foot was about 1 per cent lower than the others.

The effect of omitting either one or both of the two finest sizes in the aggregate without altering the gradation in any other way is shown in the last part of Table I. The omission of fines and consequent increase in fineness modulus permitted a slightly lower water-cement ratio to be used, and slight reductions in shrinkage resulted. Had the fineness modulus been increased by equivalent amounts without omitting fines, similar reductions in shrinkage would have been expected. Within ordinary limits, the amount of fines appears to have little effect on shrinkage.

The minimum possible shrinkages with a given cement and aggregate were expected to result from using the leanest mixes that could be placed without excessive air voids. Such mixes are represented in Table II, which gives shrinkage results on a variety of gradations with barely enough cement to keep the air voids from exceeding 2 per cent with a slump of 3 in. The slump could not be measured accurately in some of the unusual mixes, and therefore the water-cement ratios are not always comparable. All mixes could not be said to be of equal workability.

In the first part of Table II, results are given for incomplete gradations differing from one another only in minimum sizes of aggregate. The mixes necessary to keep the air voids below 2 per cent varied from 1:8.5 for the complete mix to 1:2.5 for a minimum size of $\frac{3}{8}$ in. Water-cement ratios varied from 0.77 to 0.36, respectively. In spite of the great range of mixes and minimum sizes of aggregate, the shrinkage varied but slightly except for those gradations that contained no aggregate passing the No. 4 sieve. Even when the aggregate was all between $\frac{3}{8}$ and $\frac{3}{4}$ in., the shrinkage was only 22 per cent less than for the complete gradation (No. 1).

In the second part of Table II are a few results on gap gradations, where some intermediate sizes of aggregate are omitted. Again, barely enough cement was used to keep the air voids

TABLE III.—EFFECT OF AGGREGATE ON SHRINKAGE OF CONCRETE OF FIXED CEMENT CONTENT.

Type and Origin of Aggregate	Water-Cement Ratio, by Weight	Specific Gravity of Aggregate	Absorption of Aggregate, per cent	6-month Shrinkage, millionths		
				2-day cure (observed)	28-day Cure	
					Observed	Corrected for Water-Cement Ratio[c]
Natural Sands and Gravels:						
Sandstone[a] (Ohio River)	0.65	2.52	3.8	860	810	810
Siliceous, mixed (Mass.)	0.65	2.74	1.0	710	700	700
Siliceous, mixed (Ohio River)	0.70	2.58	2.2	610	670	610
Dolomite pebbles, crushed sand (Wis.)	0.78	2.68	1.7	560	590	430
Siliceous and calcareous, mixed, Boulder Dam (Ariz.)	0.62	2.63	1.3	570	540	570
Mixed coarse aggregate (Mass.), spherical slag sand[b]	0.67	....	....	540	530	510
Largely quartz (Conn.)	0.63	2.66	0.8	....	350	360
Crushed Aggregates:						
Crushed mixed gravel (Mass.)	0.87	2.74	1.0	....	870	520
Serpentine marble (Vermont)	0.82	2.78	0.3	760	830	550
Marble, pure (Vermont)	0.84	2.77	0.3	650	670	420
Hornblende (Canada)	0.69	3.28	0.4	....	650	590
Pyroxene (Canada)	0.65	3.51	0.4	....	630	630
Granite (Mass.)	0.80	2.67	0.7	510	590	410
Dolomite (Tenn.)	0.80	2.80	0.3	550	500	350
Feldspar (Va.)	0.80	2.58	0.2	....	480	330
Dolomite (Va.)	0.73	2.80	0.2	460	450	370
Limestone (Calif.)	0.77	2.74	0.2	470	450	330
Quartz (New York)	0.79	2.65	0.1	....	450	320

[a] Coarse aggregate only consisted of sandstone pebbles hand picked from Ohio River gravel.

[b] Spherical slag sand artificially made.

[c] Shrinkages corrected to water-cement ratio of 0.65 by factor of 1.75 per cent change in shrinkage for each 1 per cent difference in water content.

NOTE.—Cement content, 1.30 bbl. per cu. yd.; slump 3 in.; standard cement A used throughout.

below 2 per cent with a slump of 3 in. An appreciable reduction in shrinkage, up to 23 per cent maximum, is shown for the leanest mixes, in which the water-cement ratios were over 1.0 by weight. Except for these impractical mixes, no great improvement is indicated for gap gradations with this type of aggregate.

Possible Reason for Slight Effect of Gradation of Aggregate:

The particular kind of cement and aggregate used in the gradation studies reported above may explain the slight variation that was found. The cement was a standard brand that gave fairly high shrinkage without great tendency toward cracking. Likewise, the aggregate was one that gave high shrinkage in spite of a rather high rigidity of particles and apparently did not encourage cracking between particles. If no cracking occurred between aggregate particles, the results are about as might be expected, because there was little difference in water volume in the various mixes. But if extensive cracking occurred, the uniformity of results is not readily explained. At any rate, it should not be concluded that similar results will necessarily be obtained with all materials. Corresponding tests are now in progress on other types of cement and aggregate.

Effect of Type of Aggregate on Shrinkage:

Large differences in concrete shrinkage are obtained from different aggregates, especially in lean mixes. Some difference results from the fact that one aggregate requires more mixing water than does another, but the greater difference is probably due to the physical properties of the aggregate itself. In the following table are comparative shrinkages for concretes of identical water-cement ratios (0.40) and mix (1:2.5), containing only a single size of aggregate ($\frac{3}{16}$ to $\frac{3}{8}$ in.). The secondary differences that are usually present in concretes containing different aggregates are here avoided and the effect of the aggregate alone is shown. It may be noted that a range of shrinkage from 320 to 680 millionths is shown for concrete containing different types of pebbles hand picked from the same common gravel. Also, a still larger range is found when the aggregate (sandstone) giving the highest shrinkage, is compared with those giving low shrinkage. In the low-shrinkage class are limestone, quartz, and glass. Many other materials, such as good grades of dolomite and granite, also fall in this class but are not included in the table for lack of comparable data.

EFFECT OF TYPE OF AGGREGATE OF A SINGLE SIZE ON THE SHRINKAGE OF CONCRETE.

Aggregate	Specific Gravity	Absorption, per cent	1-yr. Shrinkage, millionths
A—mixed gravel (Mass.)	2.74	1.0	560
B—hand-picked from A (slate pebbles)	2.75	1.3	680
D—hand-picked from A (granite)	2.67	0.8	470
E—hand-picked from A (quartz pebbles)	2.66	0.3	320
F—sandstone (West Virginia)	2.47	5.0	1160
G—solid glass spheres	....	0	250
H—crushed limestone (Calif.)	2.74	0.2	410

In Table III, results are shown for a large number of aggregates for full concrete mixes with water-cement ratio adjusted for fixed consistency. The results are separated into two groups according to whether or not the aggregate is "sand and gravel" or "crushed," and are arranged in order of decreasing shrinkage. Two ages of preliminary moist curing were employed, 2 and 28 days. Most of the crushed aggregates were tumbled in a ball mill before using to improve the particle shape and thus prevent the need for a very high water content. The dolomites were exceptions, as they were received from aggregate plants designed to furnish favorable particle shape. There remained a considerable amount of variation in water-cement ratio for different aggregates despite the milling operation. The extent of improvement is indicated by the fact that for the granite aggregate a water-cement ratio of 1.04 was required before milling, as compared with 0.80 after milling.

The effect of variable water-cement ratio on the shrinkage of concretes containing different aggregates has been eliminated by empirical corrections to obtain the values in the last column of Table III. No great reliability can be expected for the corrections, which amount to almost 2 per cent for each 1 per cent of water-cement ratio above or below 0.65, because the same factor was applied regardless of the type of aggregate. Insufficient data were available for greater refinement. The corrected shrinkages, however, are believed to be fair indications of the ability of each type of aggregate to restrain the shrinkage of a given cement paste.

A few interesting revelations can be noted from the results, although a detailed analysis will be postponed until a later date when tests on the governing properties of the cement paste and aggregates are completed. Among the pure-mineral aggregates included in Table III, pyroxene and hornblende are alone in producing high shrinkage of concrete. On the other hand, quartz, feldspar, limestone, and dolomite are outstanding in producing low shrinkage. Granite, being a combination of various minerals, logically falls intermediate between the groups producing high and low shrinkage. Granites of higher rigidity would be expected to show lower shrinkages, and *vice versa.* Likewise, limestones and dolomites cover a wide range of rigidity and those represented are believed to be of greater-than-average rigidity. The only data available on the pure marble indicate it to be more readily compressible than the limestone; this might explain the higher shrinkage for marble concrete. From studies to date, it must tentatively be concluded that compressibility is the most important single property of aggregate governing concrete shrinkage.

Among the aggregates of mixed composition, those producing concrete of high shrinkage often contain a large amount of sandstone, slate, or other particles which shrink of themselves or have low rigidity. The mixed gravel from Massachusetts was a glacial gravel containing much slate and granite, along with a moderate amount of quartz, all having appreciable porosity. Conversely, those producing concrete of low shrinkage often contain large amounts of quartz, limestone, feldspar, or other particles of high ridigity. Sandstone and slate cause high shrinkage not only because of their compressibility but also because these aggregates themselves will generally contract appreciably upon drying. The shrinkage due to drying from a saturated state of particles of slate from the mixed gravel is over 200 millionths. Particles of all other aggregates tested, except the sandstone, have low shrinkage.

Particle shape was found to have little effect on concrete shrinkage unless advantage was taken of the lower water requirement of well-shaped aggregates. Crushed dolomite particles were rubbed down to the shape of natural pebbles by extended grinding in a ball mill. Concrete made from these pebbles exhibited nearly the same shrinkage as concrete of the same water-cement ratio containing ordinary crushed dolomite. Conversely, smooth gravel was crushed and tested in comparison with the uncrushed gravel. When the water-cement ratio was the same, no difference in concrete shrinkage was observed.

Continued tests are expected to evaluate more adequately the effect of (1) compressibility of aggregate and extensibility of cement paste, (2) cracking between particles, (3) contact between paste and aggregate, and (4) volume change of the aggregate itself. The inherent shrinkage of the cement paste is being determined on specimens of various water-cement ratios and of such thin dimensions as to be free from cracking. In determining the compressibility of aggregate, plastic flow is included by measuring deformations under sustained load, because, when concrete shrinks, the compression of aggregate particles is gradual and stress is sustained over long periods of time. Likewise, in determining extensibility of cement paste, the modulus of elasticity determined by ordinary, quick-loading tests is of little value. It is indicated, for example, that with a modulus of elasticity of 4,000,000 lb. per sq. in. determined in the usual test, the sustained modulus of elasticity for continued loading may be less than 1,000,000 lb. per sq. in. Thus the extensibility may be more than four times that indicated by the ordinary test.

Effect of Size of Aggregate:

It was shown above that under the conditions of fixed water content and no cracking between aggregate particles, size of aggregate does not affect concrete shrinkage. But under practical conditions, a large reduction in shrinkage is obtained for larger maximum sizes of aggregate, both because of the lower water contents that can be used and because the larger aggregate sizes encourage cracking between particles.

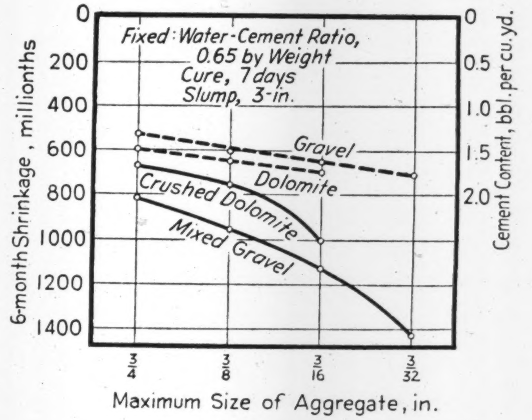

FIG. 3.—Effect of Aggregate Size on Shrinkage of Concrete of Fixed Water-Cement Ratio but Variable Mix.

In Fig. 3 are shown the shrinkages of concretes containing different maximum sizes of two types of aggregate, crushed dolomite and mixed sand and gravel. The gradations of aggregate were made comparable by employing "ideal" gradations in which the amount of each successively smaller size was a definite percentage of the coarser size. For the natural aggregate, this percentage was 70, and for the crushed dolomite it was 80, these percentage gradations giving appropriate fineness moduli for every mix represented in Fig. 3.

In obtaining the curves of Fig. 3,

both water-cement ratio and consistency were maintained constant. A richer mix was therefore required for the crushed dolomite concrete. It is interesting to note that this richer mix reduces somewhat the large advantage of low shrinkage otherwise held by the crushed dolomite. Also, the dolomite shows a greater increase in shrinkage with decreasing size. The increases in shrinkage due to reducing the maximum size of aggregate from $\frac{3}{4}$ to $\frac{3}{16}$ in. were 50 per cent and 40 per cent for crushed dolomite and mixed gravel, respectively. Thus, if a concrete mix were desired that would provide a sensitive comparison of aggregates, it probably would be one of low cement content and large aggregate size.

Effect of Composition of Cement on Concrete Shrinkage:

It has been shown that when tested neat the three main compounds in cement line up in order of decreasing shrinkage as follows: (1) tricalcium aluminate (C_3A), (2) dicalcium silicate (C_2S), and (3) tricalcium silicate (C_3S). Likewise in concretes, when these pure compounds are used in place of cement, the shrinkages are in the same order. Exceptions are concretes containing C_3A and C_2S under long-continued drying. The C_3A concrete usually cracks almost immediately upon commencement of drying and although high shrinkage is evidenced by the extent of cracking, measured values of over-all length contraction may not be large. Concrete containing pure C_2S is prone to crack, but if the moist curing period is long or the drying is slow, cracking will be minimized. The following results are believed to be representative comparisons of the relative behavior of C_3S, C_2S, and standard cement in concretes of fixed cement content and slump. Some cracking was suspected in all specimens, but none was visible:

	Aggregate	Water-Cement Ratio	3-month Shrinkage, millionths
Standard cement A.....	Crushed dolomite	0.80	530
Dicalcium silicate.....	Crushed dolomite	0.80	740
Tricalcium silicate.....	Crushed dolomite	0.73	430

Tests on laboratory-made cements, in which composition is the only variable, have indicated the compounds to contribute to shrinkage of concrete in the order named above. But after testing a great many commercial cements from scattered plants, one must conclude that the shrinkage characteristics of a cement cannot be predicted reliably from the ordinary analysis. The percentage of C_2S most often proves to be a good guide, but the percentage of potential C_3A does not seem to bear a consistent relation with the shrinkage performance. Great strides are being made in taking account of volume change due to free lime and magnesia, but still other factors are involved. Occasionally, a cement is encountered that is "out of line" and whose shrinkage cannot be explained by known elements of composition. For example, cement N, mentioned above, is a cement that gives lower shrinkage than other cements of similar analysis. Probably some important feature of the composition is being neglected. The most enlightening studies now in progress are expected to be those aimed to determine: (1) the true, rather than potential, compositions, (2) the effect of glass content, (3) effect of alkalies, and (4) the effect of the inert form of C_2S acting as an adulterant that may reduce the amount of active C_2S. In view of the unsatisfactory present status of studies of shrinkage as affected by features of composition not already discussed, detailed analysis will not be attempted herein.

Comparisons of shrinkage results for concretes containing selected cements and aggregates are presented in Table IV. It may be noted that there is a range of as much as 2 to 1 in the relative shrinkages exhibited by different cements with the same aggregate. Furthermore, there is a still greater range when both different cements and different aggregates are considered. It is hardly conceivable that the inherent shrinkage of the cement pastes contained in the concretes varied by such large amounts. Nor is it readily conceivable that the restraining influence of the aggregate could account for the large variations without admitting internal cracking to be a factor.

TABLE IV.—SHRINKAGE OF CONCRETE CONTAINING DIFFERENT CEMENTS AND AGGREGATES.

Aggregate	Cement Brand[a]	Shrinkage Characteristic of Cement	Water-Cement Ratio, by weight	6-month Shrinkage, millonths	
				After Moist Curing 2 days	After Moist Curing 28 days
Mixed gravel........	A	Normal	0.65	820	810
	N-2	Low	0.63	610	620
	N	Very low	0.62	474	470
Crushed dolomite...	A	Normal	0.80	550	496
	N-2	Low	0.76	400	430
	N	Very low	0.79	400	320
Crushed marble.....	A	Normal	0.84	650	660
	N-2	Low	0.80	430	350
	N	Very low	0.81	260	320
Crushed granite....	L	High (low heat)	0.87	550	800
	A	Normal	0.87	730	680

[a] Potential composition of cements:

	C_3S	C_2S	C_3A	C_4AF
A..............	51	18	15	7
N-2............	59	13	10	8
N..............	52	24	6	12
L..............	38	33	5	15

The effect of duration of preliminary moist curing is also shown in Table IV. In some cases, a 28-day cure produced less shrinkage than did a 2-day cure, but in about as many cases the effect was opposite. The behavior of low-heat cement with crushed granite aggregate is of interest. With only 2 days of preliminary curing, the shrinkage was relatively low, while with 28 days of moist curing, the shrinkage was almost the maximum shown. A likely reason is that the low-heat cement develops but little strength at 2 days and the aggregate can effectively restrain the weak paste from shrinking. After the strength that comes with 28 days of curing, however, the large inherent shrinkage of the low-heat cement manifests itself. Again, cracking seems to be necessary for the full explanation of the unusually low shrinkage with 2 days of curing. But the difference in extensibility of the cement pastes for the two ages of curing is in the right direction and may be sufficient to account for the large difference in shrinkage.

TABLE V.—SHRINKAGE OF CONCRETES CONTAINING FRACTIONATED CEMENTS.

Type of Clinker	Range of Particle Size, μ	Fineness, sq. cm. per g.	Potential Composition, per cent			Water-Cement Ratio, by weight	1-yr. Shrinkage, millionths
			C_3S	C_2S	C_3A		
Low C_3A........	0 to 5	4095	65	11	2.7	0.70	580
	5 to 10	2945	64	13	2.2	0.73[a]	595
	5 to 48	1600	58	21	2.5	0.58[a]	468
	10 to 17	1350	60	20	2.7	0.57[a]	534
	30 to 48	475	51	28	2.6	0.52[a]	543
High C_3A.......	0 to 5	5430	58	16	15.7	0.88	1380
	30 to 48	490	50	25	15.6	0.52[a]	611

[a] Concrete bled considerably; true water-cement ratio undoubtedly lower. Specimens are 1½ by 1½ by 12-in. bars of concrete. Aggregate is mixed gravel, ¾-in. maximum size. Preliminary moist curing 28 days.

Effect of Fineness of Cement:

Although finer cements generally result in greater concrete shrinkage, the increase in shrinkage with increasing fineness is not large, and in some cases, finer cements produce concretes of lower shrinkage. Here again, there are probably two opposing influences. Finer cements hydrate more extensively and thus produce a denser gel, which probably has a lesser desired shrinkage. But the gel from the finer cement is also stronger and therefore it can realize against restraint of aggregates a larger percentage of its desired shrinkage. Whether or not increased fineness will cause increased shrinkage may depend upon which of these influences is greater. With one cement composition and type of aggregate, increased fineness may result in greater shrinkage, while under other conditions the reverse may be true.

An examination of the shrinkage results on air-separated cements covering an abnormally large range of fineness may well prepare the mind for the more practical comparisons. In Table V are the results on so-called "fractions" of cement, in which the particle-size range has been limited by dry-air separation. Referring to Table V, it may be noted that, on the average, the low-aluminate cement shows no increase in concrete shrinkage for the finer fractions. In fact, if correction is made for variations in water-cement ratio, a lower concrete shrinkage is indicated for the finer fractions. In view of the tremendous range of fineness represented in Table V, these results are impressive.

Turning to the high-aluminate cement, for which values are also shown in Table V, a different trend is indicated. For this composition, a rather large increase in shrinkage is shown for the finer of the two fractions listed. For a ratio of specific surface areas of more than 10 to 1, the corresponding ratio of shrinkages is a little more than 2 to 1. In view of the facts that a part of the difference in shrinkage may be attributed to difference in water contents and that the fineness range is enormous, the effect of fineness is not alarming, even for this composition.

More or less in accordance with

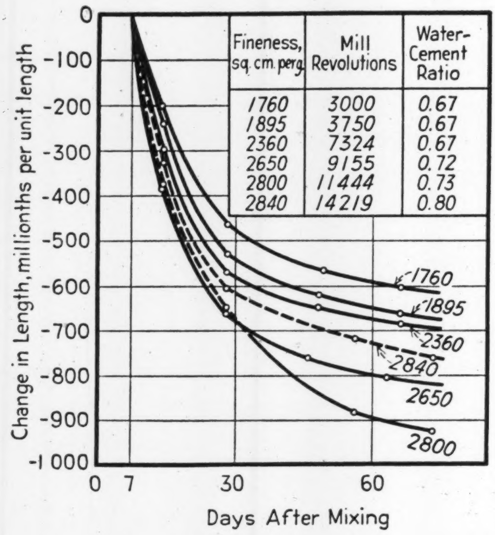

Fig. 4.—Effect of Fineness of Cement on Shrinkage of Concrete.

the shrinkage results for fractionated cements, standard cements of low C_3A content usually show but little change in concrete shrinkage with increasing fineness. Cements containing a fair amount of C_3A, however, may exhibit seriously-high shrinkage if ground very fine. In Fig. 4 are shown the measured shrinkages of concretes containing cements of identical composition but differing in fineness over a range entirely above that of standard cements. The features of the composition were 10 per cent of potential C_3A and 39 per cent of C_3S. Referring to Fig. 4 it may be noted that there is a progressive increase in concrete shrinkage with increasing fineness of cement except for the cement of highest fineness. The latter cement may have been altered physically by "overgrinding," which is evidenced by the fact that almost no increase in fineness resulted from the final 2800 revolutions of the mill. That this cement had different properties from the others is indicated by its excessive water requirement (water-cement ratio = 0.80). Excepting this cement, the curves of Fig. 4 present a fairly clear picture of the increasing shrinkage of concrete with increasing fineness of cement. Further study is being devoted to overgrinding.

Effect of Gypsum Retarder:

Tests were made on two compositions of clinker, one of high and one of low C_3A content, to determine the effect of different amounts of gypsum. To make one pair of cements, each clinker was ground to a specific fineness with barely enough gypsum to pass the requirements for setting times. To make a second pair of cements, a sufficient amount of gypsum was used to bring the SO_3 content to 2.5 per cent. Considering first the composition of low C_3A content, no difference in mixing water was necessary and no difference in shrinkage resulted for 1.0 per cent of SO_3 as compared with 2.5. For the high-aluminate cement, approximately 10 per cent more mixing water was required when the SO_3 percentage was 1.3 than when it was 2.5. This is believed to be due to a tendency toward quickset with the low SO_3 content. The cement with the 1.3 per cent of SO_3 produced a concrete of 30 per cent greater shrinkage. From these and other test results, it is concluded that the amount of gypsum has little effect unless the cement may be insufficiently retarded with the smaller amounts of gypsum involved. In the latter case, the amount of gypsum is important.

Effect of Admixtures:

It was reasoned that if internal cracking between aggregate particles was a factor in concrete shrinkage, the introduction of a material to reduce the tensile strength of the mortar might reduce shrinkage. A first and unsuccessful trial consisted of adding 2 and 4 per cent, respectively, of 24-mesh ground mica to a standard cement and making concrete specimens. The mica required the use of so much extra mixing water that the concrete shrinkage was increased rather than decreased, as shown in Table VI. But the increase in shrinkage was small compared with the increase in water-cement ratio, so a further trial was made.

A more successful trial consisted of adding $\frac{1}{2}$, 1, 2, and 4 per cent, respectively, of 60-mesh mica to the same standard cement. This finer mica, weight for weight, did not increase the water requirement as much as did the coarser mica. Lowest shrinkage resulted from the addition of only $\frac{1}{2}$ per cent of the 60-mesh mica, the reduction in shrinkage at 3 months amounting to 22 per cent. No lower percentages of mica were tried and higher percentages gave less reduction in shrinkage, all as shown in Table VI. If this type of mica were to find a place in service concrete for reducing shrinkage, its effect on other properties should first be determined.

Also included in Table VI are shrinkage results on concretes containing varying amounts of a dispersing agent (calcium lignin sulfonate) and of a well-known wetting agent but of unknown composition. In the amounts used, the dispersing agent had little effect on the shrinkage or on the air-void content of the concrete. The wetting agent, however, increased the air voids appreciably and consequently permitted the use of appreciably less mixing water. The net result was that the early shrinkage was increased with increasing amounts of the wetting agent, but the later shrinkage was practically unaffected. Calorimeter tests indicated neither the dispersing agent nor the wetting agent to have a noticeable effect on hydration.

TABLE VI.—EFFECT OF ADMIXTURES ON SHRINKAGE OF CONCRETE.

All specimens contain mixed gravel of $\frac{3}{4}$-in. maximum size; cement content of 1.3 bbl. per cu. yd., and were moist cured for a period of 7 days.

Admixture, per cent by weight of cement	Water-Cement Ratio	Air Voids, per cent	Shrinkage, millionths		
			28 days	3 months	5 months
None (control)	0.71	1.6	330	650	....
2 of 24-mesh mica	0.83	2.1	305	670	....
4 of 24-mesh mica	0.93	1.7	315	670	....
None (control)	0.71	1.6	330	650	....
0.5 of 60-mesh mica	0.73	2.2	330	510	....
1.0 of 60-mesh mica	0.77	1.8	365	690	....
2.0 of 60-mesh mica	0.79	2.0	375	610	....
4.0 of 60-mesh mica	0.83	2.2	420	830	....
None (control)	0.69	2.4	350	595	680
0.05 of dispersing agent	0.68	2.3	350	615	655
0.10 of dispersing agent	0.67	2.6	340	580	630
None (control)	0.69	2.4	350	595	680
0.02 of wetting agent	0.68	5.2	395	635	685
0.04 of wetting agent	0.63	7.3	420	615	660
0.08 of wetting agent	0.60	11.3	500	645	700

TENTATIVE CONCLUSIONS

Not all of the following conclusions are new, nor are those which may be new likely to be final. While they are believed to apply for the conditions, materials and types of tests included in this paper, insufficient confirmation has yet been obtained to label them as anything but tentative.

1. The cement paste in ordinary concrete, if unrestrained by the aggregate, would shrink from 5 to 15 times as much as does the concrete.

2. Confining attention to the particular standard cement and mixed gravel employed in the main portion of the reported tests, as long as the maximum size of aggregate and consistency of concrete are not varied, neither cement content, nor gradation of aggregate, nor duration of preliminary moist curing has much effect on the concrete shrinkage.

3. Size of aggregate, below a limiting size, does not affect concrete shrinkage unless it affects the water requirement. Above the limiting size, the larger the aggregate the less the over-all shrinkage of concrete, even for a fixed water content.

4. An increase in the maximum size of aggregate permits the use of less mixing water per cubic yard of concrete and thus causes a further reduction in shrinkage.

5. Among the pure-mineral aggregates which have been tested, hornblende and pyroxene are notable in producing concrete of high shrinkage. On the other hand, quartz, feldspar, dolomite, and limestone are outstanding in producing concrete of low shrinkage.

6. Among the aggregates of mixed composition, sandstone and slate produce concrete of high shrinkage, while a dense grade of granite produces concrete of less-than-average shrinkage.

7. The relative compressibility of

aggregate particles appears to be the most important factor causing different aggregates to produce concretes of different shrinkage.

8. Particle shape of aggregate has little effect on concrete shrinkage except as it determines the amount of mixing water that must be used.

9. Each 1 per cent of added mixing water causes an increase of concrete shrinkage of almost 2 per cent when the aggregate is mixed gravel.

10. Concretes of high cement content and containing dolomite and granite aggregates (and possibly all aggregates of high rigidity), shrink more than do corresponding concretes of lower cement content. This is in contrast with concretes containing the mixed-gravel aggregate, for which the shrinkage is practically independent of cement content.

11. Of the three most important potential compounds of cement, a given amount of tricalcium silicate contributes least to shrinkage, a like amount of tricalcium aluminate contributes most, and dicalcium silicate is intermediate between the other two compounds.

12. Variations in concrete shrinkage for different commercial cements cannot be explained satisfactorily by differences in the ordinary analysis or in fineness.

13. In general, cements of higher fineness exhibit somewhat greater concrete shrinkage when preliminary moist curing is at 70 F., but cements of very low tricalcium aluminate content appear to be an exception.

14. Quantity of gypsum in cement has little effect on concrete shrinkage unless the set is incompletely retarded for the smaller amounts; in this case an insufficient amount of gypsum causes a greater water requirement and therefore a correspondingly greater shrinkage.

15. Admixtures consisting of particular dispersing and wetting agents in nominal amounts have but little effect on shrinkage of concrete. The wetting agent appreciably increases air voids and permits a substantial decrease in water-cement ratio with practically no net effect on ultimate shrinkage.

16. An appreciable reduction in concrete shrinkage can be obtained by adding $\frac{1}{2}$ per cent (by weight of cement) of commercial, ground mica of 60-mesh size.

17. Cracking of cement mortar between aggregate particles is believed to be a factor contributing to the low overall shrinkage of many concretes.

SELECTED ADDITIONAL REFERENCES

R. E. Davis, "Summary of Results of Investigations on Volume Changes in Mortars and Concretes, Due to Causes Other than Stress," *Proceedings*, Am. Concrete Inst., Vol. 26, p. 407 (1930).

R. E. Davis, R. W. Carlson, G. E. Troxell and J. W. Kelly, "Cement Investigations for Boulder Dam," *Proceedings*, Am. Concrete Inst., Vol. 30, p. 485 (1934).

F. R. McMillan, "Volume Changes of Concrete," *Proceedings* (London Congress), Internat. Assn. Testing Mats., p. 401 (1937).

Y. Yoshida, "Length Changes of Cement Paste in Relation to Combined Water," *Proceedings*, Am. Concrete Inst., Vol. 9, p. 25 (1937).

DISCUSSION

Mr. T. C. Powers.[1]—We are all very much interested in the problem of cracking and shrinkage, and the paper presented by Mr. Carlson is a very valuable contribution to it. There is one point which I think might bear some reconsideration. On the whole, it is easy to follow Mr. Carlson's reasoning, but there is one inference to the effect that if the aggregate particles exceed a certain size it is almost certain that cracks will develop around them. Such cracks might develop, but I do not believe that the experiment reported definitely proves it. The experiments made were something like this: Cement pastes of a creamy consistency (40 per cent mixing water) were made, and to these were added aggregate particles of different sizes. It was found that when the particle size exceeded a certain limit, the over-all shrinkage of the specimen dropped off sharply. The cause of that reduction in shrinkage was believed to be the formation of cracks around the aggregate particles, thereby relieving the stresses which would have caused a contraction of the entire specimen. It seems possible to account for this reduction in shrinkage in another manner. We know that particles do not readily settle through a paste of the consistency used. A particle not too large would remain almost permanently suspended, since it takes a certain minimum force to cause the particle to fall through a paste of that consistency. In other words, it is probable that in the specimens made with the smaller particles, the particles remained suspended in the paste, not in contact with one another. But, when the larger particles were used, they settled into contact with one another before the paste congealed. Thus there would be, through the body of the paste, a rigid framework which would naturally restrain the shrinkage of the paste more than an equal volume of small particles not in contact with one another. In this connection, it should be noted that mixes made with graded particles did not indicate the strong reduction in shrinkage that the experiment with the large single-sized particles did.

Mr. Roy W. Carlson.[2]—Mr. Powers' points are all well taken. The cracking in the cement paste between the aggregate particles is not conclusively proved by the tests we have presented; I agree with Mr. Powers that we should not exclude any possible explanation. In future work, we expect to modify our technique so that we will get the intrinsic shrinkage of the cement paste and not the shrinkage of the fairly large specimens as affected by stress and cracking.

Mr. J. C. Pearson.[3]—I understand from the paper that the localized cracking of the cement paste would be more apt to occur in concrete made with the more rigid aggregates, that is, in concrete which shows low over-all shrinkage. If this is the case, would not one infer that this type of concrete should be more vulnerable to attack by freezing and thawing?

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[2] Associate Professor of Civil Engineering, Massachusetts Institute of Technology, Cambridge, Mass.

[3] Director of Research, Lehigh Portland Cement Co., Allentown, Pa.

MR. CARLSON.—That is a rather delicate subject. I think by all means our attempts should be to make the intrinsic shrinkage of the cement paste or mortar as low as possible, and beyond that point, if we can gain a low over-all shrinkage by encouraging the formation of internal cracks which are so small as to be invisible, my own opinion is that this might be desirable. In any event, the first aim should be to get a low intrinsic shrinkage of the mortar or paste.

MR. PEARSON.—Mr. Carlson has not given a direct answer to my question, that is, whether low shrinkage concrete would be expected to be more susceptible to damage from freezing and thawing.

MR. CARLSON.—Yes, it would.

MR. F. H. JACKSON.[4]—I was very much interested in what Mr. Carlson said regarding the effect of the physical characteristics of the aggregate on drying shrinkage. I think his tests may go far to explain certain observations we have made in our laboratory in connection with our tests of blended cements. The results of these tests are presented in the paper by Kellermann and Runner.[5]

Mortar specimens containing two entirely different types of sand, one essentially calcareous and the other practically a pure quartz sand, behaved quite differently when subjected to an accelerated weathering test which included a drying cycle. In the former case we obtained a very high residual shrinkage, whereas with the quartz sand we noted very little permanent shortening at the conclusion of the cycle. I have been searching for an explanation of this and it is quite possible that Mr. Carlson's theory will cover it.

MR. THADDEUS MERRIMAN.[6]—It would be well in all investigations of this kind to give more attention to the cement used and to record its characteristics completely at the time when the tests are made, so that later on the results can be coordinated with other tests made with other cements. The cement used in such investigations is referred to by number only. Its composition, analysis and alkali content, together with the other factors which go toward influencing its shrinkage are not stated. For some strange reason no investigator refers to the cement he uses by name of the plant of its origin. The identity of the cement is kept secret with the result that his observations cannot be correlated with those of other investigators.

Because of this condition, the literature is filled with the results of tests which conflict and vary the one from the other. In no other branch of scientific endeavor are the characteristics of the materials used kept so hidden from view. In the case of portland cement it is of especial importance that its history should be completely disclosed because upon that history depend its characteristics and its behavior. A thoroughly calcined clinker, freshly ground without water, makes one kind of cement. An underburned clinker, aged for months out in the weather and ground with water, makes another kind. Between these extremes all possible variations occur. The experimenter ought to know the characteristics of the cement he uses.

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[4] Senior Engineer of Tests, U. S. Bureau of Public Roads, Washington, D. C.

[5] W. F. Kellermann and D. G. Runner, "The Effect of Using a Blend of Portland and Natural Cement on Physical Properties of Mortar and Concrete," see p. 329.

[6] Consulting Engineer, Board of Water Supply, City of New York, New York City.

STUDIES OF THE OPERATION OF THE WAGNER TURBIDIMETER

By H. S. Ponzer[1] and D. R. MacPherson[1]

Synopsis

A summary is given of data accumulated over a period of years on the operation of the Wagner turbidimeter. Most of the studies reported were made on various factors of operation with the object of perfecting testing technique and establishing conditions of operation which would give better concordance and reliability of results.

The data presented indicate that inability to obtain a high degree of concordance among repeated tests is mainly the result of failure to establish sufficiently rigid technique and uniform conditions of test. With careful attention to these, the turbidimeter appears capable of giving results falling within about ±1 per cent of the average.

Data are also included from tests designed to study the fundamental reliability of the instrument. A new method of standardization is described.

The Wagner (**1**)[2] turbidimeter has been in almost constant use by the Portland Cement Association for over four years, its major use having been in connection with various clinker grinding investigations. Several thousand determinations of specific surface have been made on samples of widely differing fineness, ranging from the very coarse product of the impact grindability test (**2**) to extremely fine cement.

Cements, intermediate products, and clinker crushed in the impact grindability test are all tested with the turbidimeter. Any inaccuracy or failure to obtain concordant data has an important effect on calculations of mill efficiency. Numerous studies have therefore been made of factors suspected of affecting the operation of the instrument with the object of improving its performance. Some data also have been accumulated on the general reliability of the instrument. This paper summarizes these tests and observations. Modifications in testing technique are discussed in connection with the comments on the various operations.

General Reliability of the Instrument

Wagner (**1**) cites data showing fairly close agreement among the results obtained with the microscope, air analyzer, turbidimeter, and other sedimentation apparatus. Tests at the Portland Cement Association also support the fundamental reliability of the instrument. In previous studies (**3**) the results obtained with the turbidimeter have been shown to be in general agreement with those of the Pearson type air analyzer (**4**). The differences between the specific surface values obtained by the two methods appeared to be almost entirely due to differences in the assumptions made as to the average size of the minus 10-μ material and not to fundamental differences in the two instruments.

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[1] Manufacturing Research Engineer, and Assistant Engineer, respectively, Manufacturing Research Bureau, Portland Cement Assn., Chicago, Ill.

[2] The boldface numbers in parentheses refer to the reports and papers given in the list of references appended to this paper, see p. 455.

Tests on carefully prepared fractions, each covering a narrow size interval give further indication that the turbidimeter "sees" the various particles and is capable of measuring their surface, thereby supplementing the previous work of Wagner. The following tabulation gives the surface areas of a number of these fractions as determined by the microscope and the turbidimeter:

Nominal Size of Fraction, μ	Average Size (Microscope), μ	Surface Area, sq. cm. per g.	
		Microscope	Turbidimeter
0 to 5	3.5	5430	4700
5 to 10	7.1	2690	2780
10 to 16	12.5	1525	1415
16 to 20	18.9	1005	930
20 to 30	26.7	710	710
30 to 48	38.9	490	515
48 to 74	60.0	315	330[a]

[a] Includes surface area of material coarser than No. 325 sieve

Except for the 0 to 5 μ fraction, the surface area is seen to be substantially the same by both methods. In the case of the 0 to 5 μ fraction, all of the material was finer than the smallest particle-size reading made on the turbidimeter. This and the unusual gradation of the material gave an unduly low transmittancy constant. Using a transmittancy constant equal to the average of all the determinations, the surface area was 5730 sq. cm. per g., which is in better agreement with the microscope data.

A number of residues from separations made with the Pearson type air analyzer (4) have been subjected to turbidimeter tests. In each of these a very rapid increase in the microammeter reading occurred at the time interval for the reading at the micron size at which the separation was made. Within a few seconds, the equivalent of slightly less than 100 microamperes was reached, and movement of the needle ceased, indicating that the settlement of the particles in the turbidimeter tank was in accordance with well established laws. In addition, weight distribution curves calculated from the turbidimeter tests were almost exactly coincident with the distribution curves as determined by the air analyzer.

These instances of close agreement among the results obtained with the microscope, air analyzer, turbidimeter, and other sedimentation devices are strong indications of the general reliability of the results obtained with the turbidimeter.

STANDARDIZATION OF INSTRUMENT

When the instrument is placed in operation, the light intensity must be adjusted to give a 100-microampere reading through the tank filled with clear kerosine. The use of a standard sample as specified in A.S.T.M. Tentative Method C 115 - 34 T[3] for establishing this intensity has numerous disadvantages. The introduction of an empirical factor, such as a standard sample, impairs the scientific background of the instrument. Further development of the instrument through studies by various users is hampered and discouraged by the lack of a completely scientific technique. The specific surface of 1910 which has been assigned to the standard sample, 114-B, is recognized as an approximation and probably is not a true measure of the surface, since there is no known method today, the microscope included, which can be said to measure absolutely correctly the surface area of a cement sample. In addition, any error in the testing technique is immediately and permanently embodied in the reference reading when a specific surface determination with the instrument itself is employed in the standardization.

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[3] Tentative Method of Test for Fineness of Portland Cement by Means of the Turbidimeter (C 115 - 34 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 777 (1935); also 1937 Book of A.S.T.M. Tentative Standards, p. 466.

A method of standardization along scientific lines involving only microampere readings through clear kerosine has been devised which appears to insure the establishment of the proper light intensity to give a 100-microampere reading. When using this method, the amber filter is temporarily replaced by a piece of black paper, held between two glass plates, in which a small slit (7 by 28 mm.) has been cut to allow a beam of light to be transmitted to the photoelectric cell. Repeated microampere readings with and without the slit filter in the light path are then taken at several light intensities, none of which exceeds 50 microamperes when the slit filter is not in the light path. The ratio of the readings at each light intensity measures the light-retarding property of the slit filter. Since this ratio has been found to be practically constant over a wide range of light intensities, the light intensity can be adjusted to give a reading through the slit filter of less than 50 microamperes, which is equivalent to a 100-microampere reading with the filter removed. After making this adjustment the slit filter is replaced by the amber filter and the permanent reference reading recorded. Table I is a typical example of the calibration of the instrument using the slit-filter method.

TABLE I.—STANDARDIZATION OF TURBIDIMETER BY THE SLIT-FILTER METHOD.

Each reading taken 5 min. after filter was introduced in or removed from path of light.

Test	Microampere Readings		Ratio of Readings, (1) to (2)
	Through Slit Filter (1)	Slit Filter Removed (2)	
1	11.50	25.45	0.452
2	14.85	32.60	0.456
3	17.50	38.60	0.453
4	19 90	44.00	0.453
5	22.00	48.70	0.452
6	20.50	45.50	0.451
7	18.50	41.10	0.451
			Avg. 0.453

Adjusting the light intensity to 45.3 microamperes through the slit filter (equivalent to 100 microamperes with filter removed) gives a permanent reference reading through amber filter of 18.6.

TABLE II.—EFFECT OF TEST TUBE SIZE.

Percentage passing No. 325 sieve, 90.8; 0.4-g. sample for all tests.
Diameter of brush, 0.750 in.

Test Tube Size, in.	2 − log I		Ratio, $\frac{2 - \log I_{7.5}}{2 - \log I_{60}}$	Log Summation	Transmittancy Constant (1 g.)	Specific Surface, sq. cm. per g.	
	60 μ	7.5 μ				Individual Transmittancy Constant	Average Transmittancy Constant
VARIABLE BRUSH SPEED AVERAGING APPROXIMATELY 2500 R.P.M.							
0.844	0.9208	0.6216	0.675	1.7592	784	1810	1760
	0.9101	0.6021	0.661	1.7852	773	1760	1740
0.875	0.9318	0.6308	0.677	1.8409	750	1750	1780
	0.9101	0.5986	0.659	1.8330	753	1710	1740
0.900	0.9136	0.6289	0.689	1.7732	778	1780	1750
	0.9172	0.6055	0.660	1.8267	755	1730	1750
0.938	0.9172	0.6216	0.678	1.7676	780	1790	1750
	0.9136	0.6021	0.659	1.8501	746	1710	1750
Avg.	0.9168	0.6139	0.670	1.8045	765	1750	1750
CONSTANT BRUSH SPEED OF 3500 R.P.M.							
0.844	0.9586	0.6536	0.682	1.8268	755	1810	1830
	0.9355	0.6345	0.678	1.7960	768	1800	1790
0.875	0.9355	0.6383	0.682	1.7842	773	1810	1790
	0.9468	0.6478	0.684	1.8269	755	1790	1810
0.900	0.9393	0.6402	0.682	1.7994	767	1800	1800
	0.9355	0.6364	0.680	1.7954	768	1800	1790
0.938	0.9431	0.6402	0.679	1.8106	762	1800	1800
	0.9431	0.6478	0.687	1.7882	772	1820	1800
Avg.	0.9422	0.6423	0.682	1.8034	765	1800	1800

The slit-filter method is now used exclusively by the authors in standardizing the instrument. Occasional tests are run on the standard sample, in which the specific surface is usually found to be slightly less than 2000, although the results on samples taken from different vials are sometimes consistently high or low. While this is some indication of non-uniformity in the samples, a variation in the amount of dispersion of the flakes in the sample may account for some or all of the differences observed.

FACTORS AFFECTING DISPERSION

It is the authors' belief that dispersion of the sample is one of the most important parts of the testing technique. No systematic study of the numerous factors affecting dispersion has been made. The studies which have been made have been directed mainly toward solving special problems as they arose. However, the data from these studies shed much light on the dispersing operation.

TABLE III.—EFFECT OF STIRRING BRUSH SPEED.

Brush Speed, r.p.m.	Sample Weight, g.	$2 - \log I$		Ratio, $\frac{2 - \log I_{7.5}}{2 - \log I_{60}}$	Log Summation	Percentage Passing No. 325 Sieve	Transmittancy Constant (1 g.)	Specific Surface, sq. cm. per g.	
		60μ	7.5μ					Individual Transmittancy Constant	Increase
					CEMENTS FROM DIFFERENT SOURCES				
2500........	0.35	0.6756	0.3980	0.588	1.5433	83.2	717	1380	
3500........	0.35	0.6936	0.4123	0.594	1.5740		703	1390	10 (0.7%)
2500........	0.35	0.7100	0.4248	0.598	1.5736	85.4	722	1470	
3500........	0.35	0.7351	0.4516	0.613	1.5740		722	1520	50 (3.4%)
2500........	0.25	0.6675	0.4431	0.664	1.2576	80.0	604	1610	
3500........	0.25	0.6936	0.4705	0.678	1.2880		591	1640	30 (1.9%)
2500........	0.25	0.6411	0.4156	0.648	1.2166	86.8	677	1740	
3500........	0.25	0.6747	0.4542	0.674	1.2374		665	1800	60 (3.4%)
2500........	0.25	0.6768	0.4567	0.675	1.2476	91.8	699	1890	
3500........	0.25	0.6981	0.4882	0.700	1.2310		708	1980	90 (4.8%)
					CEMENTS PRODUCED FROM SAME CLINKER				
2500........	0.3	0.6392	0.4306	0.673	1.2160	78.8	739	1580	
3500........	0.3	0.6556	0.4492	0.685	1.2297		731	1600	20 (1.3%)
2500........	0.3	0.6824	0.4635	0.679	1.2533	81.6	742	1690	
3500........	0.3	0.7055	0.4882	0.692	1.2748		730	1720	30 (1.8%)
2500........	0.3	0.7558	0.5302	0.702	1.3245	85.9	740	1860	
3500........	0.3	0.7760	0.5528	0.713	1.3239		739	1910	50 (2.7%)

Effect of Test Tube Size:

Results from a short series of tests on the effect of test tube size are shown in Table II. These were made on one cement at brush speeds of 2500 and 3500 r.p.m. In the tests at 2500 r.p.m. (estimated average speed) the brushing device originally furnished with the turbidimeter was used. A new brushing device was assembled for the tests at 3500 r.p.m., which was designed to operate at constant speed. Four test tubes having diameters from 0.094 to 0.188 in. greater than the stirring brushes were used.

It is seen from Table II that within the range of sizes employed, test tube size had no appreciable effect, although the increase in brush speed from 2500 to 3500 r.p.m. increased the specific surface by approximately 50 sq. cm. The uniformly higher values of $2 - \log I_{60}$ at the higher speed is an indication of more complete dispersion. The maintenance of dispersion throughout the test, as measured by the ratio of 2 – log $I_{7.5}$ to 2 – log I_{60}, was also greater with the higher speed. The transmittancy constant was unaffected by either brush speed or test tube size.

On the basis of these tests, it would appear that the size of the test tube was of little importance. However, the cement used was produced in a mill in closed circuit with an air separator and did not contain flakes. Had flakes been present in appreciable quantities, some trend toward higher specific surfaces might have been found as the test tube size was decreased because of the greater dispersion of the flaked material.

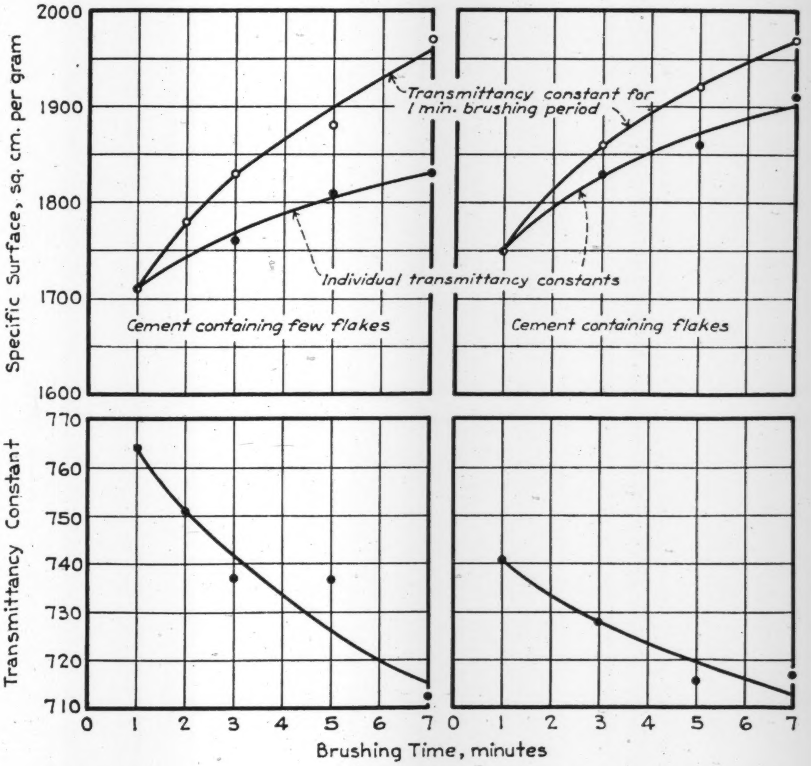

FIG. 1.—Effect of Brushing Time on Specific Surface and Transmittancy Constant.

Some users of the instrument employ test tubes of the same diameter as the stirring brush. This arrangement has the advantage of subjecting all flaked material to a vigorous brushing. Complete dispersion of all flakes which have been produced by mechanical reconsolidation of fine material during the grinding operation appears desirable because in all probability these flakes have appreciable cementing properties. In addition, better reproducibility of specific surface values is likely when all of the material is completely dispersed. If it were demonstrated conclusively that no appreciable quantity of new surface was developed from the abrasion of particles caught between the brush and test tube wall, it might be well for all users to adopt close-fitting test tubes as standard equipment.

Effect of Brush Speed and Duration of Brushing Period:

In the study of the effect of test tube size, there was indication of greater dispersion of the sample with increased brush speed with no change in transmittancy constant. The data shown in Table III give further evidence of this trend. All determinations were made with the new brushing device, constant at any speed. Increasing the brush speed from 2500 to 3500 r.p.m. was accompanied by an increase in specific surface and over-all dispersion as measured by 2 − log I_{60}. The increase in brush speed had, in general, the least effect on coarse cements and greatest on the fine. The transmittancy constants were nearly the same at both brush speeds, although there was a tendency toward slightly lower constants with the higher speed.

Retests with the brushing device operating at 3500 r.p.m., rather than 2500, have shown a similar trend toward a higher specific surface with little change in transmittancy constant. Cements free from flakes have usually shown an increase in specific surface of 3 per cent or less. With cements containing fairly large quantities of flakes, the amount of the increase has sometimes exceeded any shown in Table III. In all cases so far encountered, any increase in excess of 2 or 3 per cent has been attributed to greater dispersion of flaked material.

Results of tests with two cements showing the effect of brushing time are shown graphically in Fig. 1. Prolonging the brushing period from 1 to 7 min. resulted in substantially higher specific surfaces and lower transmittancy constants. The specific surfaces calculated from individual constants for the cement containing only a few flakes gave indication of approaching a maximum value at a brushing period somewhat in excess of 7 min., while the cement containing many flakes showed a fairly steady rate of increase in specific surface for brushing periods in excess of 3 min. With both, the increase in surface and decrease in transmittancy constant was greatest between the 1- and 3-min. brushing periods.

The increases in specific surface with increase in brush speed or brushing period are believed to be caused mainly by greater dispersion of the sample. Better dispersion of fine material in the sample, and of any flakes which may be present, could produce this effect. However, some part of the differences may be the result of the production of new surface in the brushing operation. The breaking off of corners either by impact of one particle on another or by the action of the brush on particles caught between it and the test tube wall would produce material of extremely small size. The new surface from these particles would affect the results in the same manner as greater dispersion of fine material or flakes.

Some measure of the amount of new surface which might be produced in the brushing operation may be obtained from an examination of the slope of the upper specific surface curve of Fig. 1 for the cement containing only a few flakes. These specific surfaces were calculated using the transmittancy constant for the 1-min. brushing period. If all of the increase after 3 min. of brushing was caused by the production of new surface, the average rate of increase per minute from 3 to 7 min. would be some measure of the rate of production of new surface. For this cement the rate of increase would be 35 sq. cm. per min. This represents about the maximum which could be assigned to the amount of new surface produced in the standard 1-min. brushing. Actually, better dispersion of the sample may account for part or all of the increase. It is believed, therefore, that when testing cements the production of new surface in the brushing operation is of rather minor consequence.

Recent tests have shown that under unusual conditions where large amounts of material coarser than 200 mesh are present, more than negligible quantities of new surface are produced in the brushing operation. In these tests material crushed in a grindability testing device (2) to a surface area of approximately 250 sq. cm. per g. was used. In a standard 1-min. brushing period approximately ten per cent of the surface measured was new surface produced in the brushing operation. A 2-min. brushing period at 3500 r.p.m. was found advisable for this material in order to insure adequate dispersion. The new surface created during this period of brushing was approximately 17 per cent of the apparent surface. Extensive studies have indicated that the new surface produced was caused by breakage of particles coarser than the No. 200 sieve. A method of correction employing a test on plus 200-mesh material from a parallel grindability test has been devised (3).

These tests indicate the need for further study of methods of dispersing more adequately the cement particles. In this connection it is well to mention that it has been found that oleic acid in the amount and method of use prescribed by A.S.T.M. Tentative Method C 115 – 34 T appears to give the maximum dispersion obtainable with this dispersant. However, there is still need for a more stable dispersant.

Re-use of Kerosine:

Until recently it has been the practice of the authors to re-use the kerosine a number of times, the supply being cleared after each use with activated charcoal and carefully filtered. However, comparative tests made with a fresh supply and one which had been used approximately eight times indicated that the re-use of kerosine is not advisable. In these particular tests with the re-used kerosine a sticky film was present on the walls of the settling tank (although other tests indicated that this condition did not always occur) which was not present in the tests with fresh kerosine. Presumably this was caused by the oleic acid which may have partially decomposed due to oxidation. The routine scrubbing of the inside walls of the tank after each test was sufficient to remove the cement particles, but it was impossible to remove all of the film. The presence of the sticky film on the walls of the settling tank is believed to have been the major cause of the difference between the results obtained with the new and the old kerosine supplies. The film apparently trapped fine particles which came in contact with it, thus gradually building up a layer which had a tendency to give low microampere readings, with the magnitude of the error increasing as the test progressed. As a result, the 60-μ readings were affected the least and the 7.5-μ the most. An apparent increase in the maintenance of dispersion was therefore found which reduced the log summation used in calculating the transmittancy constant. Since the log summation appears in the denominator of the equation, too high transmittancy constants were obtained.

Manipulation of Test Tube:

Stratification of the material and perhaps inadequate dispersion may occur if the test tube is held in one position throughout the brushing operation. A constant up-and-down movement of the test tube will insure a thorough mixing of the sample and allow the brush to act on all particles. Thirty of these up-and-down movements per minute with the brush held lightly against one side of the tube have been adopted as standard practice in the interest of uniformity.

Upon completion of the brushing operation, the suspension is poured into the settling tank. The test tube is held inverted above the tank and thoroughly washed with a stream of kerosine from a wash bottle (protected with a calcium chloride tube) with an upturned jet. By this procedure the transfer of the suspension is accomplished very rapidly and effectively, and with reasonable care there is little chance of any kerosine spilling on the outside of the tank. The test tube is rinsed in the same manner.

TABLE IV.—EFFECT OF AGE ON SAMPLE.

Sample weight, 0.4 g. in all tests.
Percentage passing No. 325 sieve, 90.8.

2 − log I		Ratio, $\frac{2 - \log I_{7.5}}{2 - \log I_{60}}$	Log Summation	Transmittancy Constant (1 g.)	Specific Surface, sq. cm. per g.
60 μ	7.5 μ				
FRESH SAMPLE					
0.8962	0.5836	0.651	1.8035	765	1710
0.9031	0.5836	0.647	1.7912	770	1740
0.8996	0.5884	0.653	1.7940	769	1730
Avg. 0.8996	0.5852	0.650	1.7962	768	1730
GALLON SAMPLE STORED IN SEALED CAN FOR 2 MONTHS					
0.8962	0.5817	0.649	1.8086	763	1710
0.8962	0.5800	0.648	1.7712	778	1740
0.9031	0.5952	0.659	1.7993	767	1730
Avg. 0.8985	0.5856	0.652	1.7930	769	1730
SAMPLE STORED IN 2-OZ. BOTTLE FOR 2 MONTHS					
0.8827	0.5575	0.631	1.8570	743	1640
0.8601	0.5361	0.622	1.7870	773	1660
0.8962	0.5768	0.643	1.8374	752	1690
Avg. 0.8797	0.5568	0.632	1.8271	756	1660

Effect of Age on Sample:

It is a well-known fact that cement samples deteriorate with age if exposed to the air. The data shown in Table IV are typical of turbidimeter tests with an exposed sample as compared with a fresh sample or one which has been stored in a sealed can. After two months' storage in a sealed can, the specific surface was the same as that of the fresh sample. Storage in a 2-oz. sample bottle with a tight-fitting but apparently not air-tight top permitted deterioration. The lower specific surface and greater scattering of values are characteristic of tests on such samples.

The specific surfaces of samples retested after more than a year's storage in friction-top metal cans were in close agreement with the original. However, repeat tests on fresh samples usually agree better than those made on older ones. The practice of sealing sample bottles with paraffin has also been adopted, although long-time tests have not been made to determine its effectiveness.

FACTORS AFFECTING OPERATIONS SUBSEQUENT TO DISPERSION

Kerosine Temperature:

Changes in temperature supposedly do not affect the performance of the instrument, since the timing burette is designed to adjust the rate of flow when changes in viscosity, due to temperature differences, occur. Tests have shown that the burette makes proper compensation in the range of normal room temperatures. No difficulty should then be encountered as long as the kerosine supply is exactly at room temperature. In practice this is seldom true, unless the instrument is operated in a constant-temperature room.

During a test started with kerosine of higher or lower temperature than the surrounding air, the kerosine will gradually approach room temperature as the test progresses. The rate of change in the tank and burette will not be the same, causing some error. However, a more important error arises from the setting up of currents within the settling tank. This condition is illustrated by the data shown in Table V where heating or chilling the kerosine produced substantial differences in the character of the turbidimeter results.

TABLE V.—EFFECT OF KEROSINE TEMPERATURE.

Sample weight 0.4 g. for all tests.
Percentage passing No. 325 sieve, 90.8.
Room temperature, 23.8 C. in all tests.

2 − log *I*		Ratio, $\frac{2 - \log I_{7.5}}{2 - \log I_{60}}$	Log Summation	Transmittancy Constant (1 g.)	Specific Surface, sq. cm. per g.	Temperature, deg. Cent.				Burette Time min.:sec.
60 μ	7.5 μ					Stock	Tank		Burette End	
							Start	End		
KEROSINE AT ROOM TEMPERATURE										
0.8697	0.5467	0.628	1.7781	777	1690	23.0	23.6	24.3	23.6	8:01
0.8962	0.5768	0.643	1.7920	769	1720	23.0	24.0	24.4	23.5	8:02
0.8830	0.5618	0.636	1.7850	773	1710	23.0	23.8	24.4	23.6	8:01
CHILLED KEROSINE										
0.8633	0.5482	0.634	1.8594	742	1600	11.5	17.8	21.1	20.8	8:38
0.8601	0.5272	0.613	1.8485	747	1610	14.1	17.8	20.3	20.1	8:42
0.8570	0.5302	0.618	1.8303	753	1620	16.7	19.4	20.3	20.1	8:42
0.8697	0.5452	0.627	1.8055	764	1660	20.8	22.5	23.8	23.0	8:12
0.8697	0.5452	0.627	1.8367	752	1630	21.5	23.4	24.3	23.5	8:01
0.8640	0.5392	0.624	1.8361	752	1620	16.9	20.2	22.0	21.5	8:27
HEATED KEROSINE										
0.8962	0.7235	0.807	1.5637	882	1980	46.0	37.4	31.6	29.8	6:56
0.8928	0.7447	0.834	1.4677	942	2100	53.1	42.0	35.0	30.1	6:39
0.8945	0.7341	0.820	1.5157	912	2040	49.5	39.7	33.3	30.0	6:47

Chilling the kerosine reduced over-all dispersion, maintenance of dispersion, the transmittancy constant, and the specific surface, while heating the kerosine produced the opposite effect. The difference between the extreme conditions in these tests was 420 sq. cm.

Observations on the settling of the cement particles during the test disclosed marked difference for the three conditions. With the kerosine at room temperature, the particles were observed to act as freely falling bodies in a stationary column of liquid. However, with the chilled kerosine the particles near the surfaces of the tank either settled very slowly or actually rose, while in the center the settling rate was too rapid. In this case, the walls being at a higher temperature tended to heat the nearby kerosine, causing it to rise, which in turn forced a downward movement of the kerosine in the center. With the heated kerosine, the converse was true. Direct observations and the trend of the microampere readings indicated that the rate of settlement of the particles in the center section of the column had the greatest effect. For both conditions the turbulence had the least effect on the initial reading.

At first the turbidimeter was operated in a room with no temperature control. While the data in Table V represent extreme conditions not encountered in practice, they nevertheless illustrate that differences between kerosine and room temperatures can produce errors of considerable magnitude. The instrument therefore was moved to a room in which the temperature is more uniform, which caused a general improvement in uniformity of results.

Cleaning the Settling Tank:

The settling tank must be scrupulously clean both inside and out in order to secure close duplication of results. A uniformly cloudy film over the entire area of the tank may cause no difficulty, but inspection of the tank at the completion of a test is sufficient to demonstrate that the distribution of fine particles on the sides is by no means uniform. Even after thorough rinsing, material can be seen to be clinging to certain areas. Scrubbing with a brush or a "squeegee" is a sure method of removal of such material. It can be shown that films can cause an error of several per cent in the transmittancy constant and specific surface. Previous to including in the regular test procedure the practice of scrubbing the inside of the tank, changes in the light intensity of from 0.1 to 0.3 of a microampere were required for nearly every test, but since the introduction of improved methods of cleaning the tank, changes in light intensity are seldom necessary.

Delaying Placement of Tank in Cabinet:

Delaying the placement of the settling tank in the cabinet has been suspected of being a likely source of error, since the photoelectric cell supposedly requires some time to come to equilibrium due to fatigue characteristics. However, in a series of tests in which the 60-μ reading was taken at from 11 to 44 sec. after placement of the tank in the cabinet (41 to 8 sec. for the cell to reach equilibrium), the results were nearly identical.

A more extended study might disclose certain limits of the 60-μ readings beyond which greater time would be required to allow the cell to reach equilibrium. No difficulty has ever been experienced with 60-μ readings varying from 10 to 25 microamperes, although with other photoelectric cells this range may be narrower. However, in the interest of uniformity, sample weights are now adjusted to produce initial readings within 15 to 22 microamperes, and the tank is placed in the cabinet as rapidly as possible after completing the oscillation.

Errors in Reading Microammeter:

An error in reading the microammeter in making the 60-μ reading produces a substantial difference in the transmittancy constant and hence the specific surface. While the microammeter is readable to 0.1 microampere as prescribed in the A.S.T.M. Tentative Method C 115 – 34 T the fairly rapid movement of the needle throughout the test does not permit reliance on the readings to this degree of accuracy. It is easily demonstrated that with a 60-μ reading of 10 microamperes, an error of reading of 0.1 microampere produces a 2.5 to 3.0 per cent error in the transmittancy constant and an error of 2.0 to 2.5 per cent in the specific surface. For a 60-μ reading of 20, the error is about 1.5 to 2.0 per cent for the constant and 1.0 to 1.8 per cent for the specific surface. This is strong argument for using a fixed constant for all cements of the same chemical composition and heat treatment from a given source. When this procedure can be followed, the constant should be obtained by averaging the results of a number of determinations.

325-MESH FINENESS

The results of a series of 325-mesh fineness determinations in which washing time, nozzle pressure, and sample weight were systematically varied are shown in Fig. 2. Of the three variables studied, the length of the washing period is seen to have the greatest effect. Nozzle pressure (5 to 15 lb. per sq. in.) was of minor importance. Sample size had a somewhat greater effect than nozzle pressure but was still of little significance.

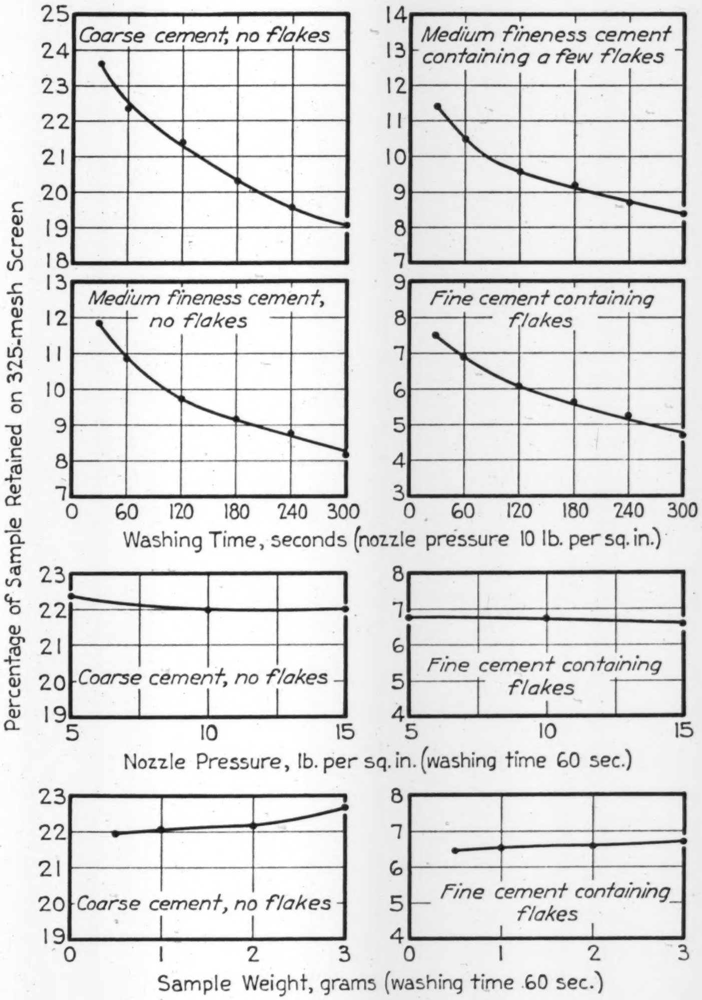

FIG. 2.—Effect of Washing Time, Pressure, and Sample Weight on 325-mesh Fineness.

The change in the slope of the curves and the trend toward lower residues as the washing period was increased are indications of the introduction of an error by adherence to a 1-min. washing period. In the absence of knowledge as to the amount of material finer than 60 μ remaining in the residue after the various washing periods, the magnitude of the possible error is not definitely known.

Presumably the change in slope of the curves in the range 30 to 120 sec. arises from failure to completely remove all of the fines and may be a measure of the total inaccuracy. The further decrease in residue weight in the range 120 to 300 sec. may be the result of further washing through of fine material. However, the nearly constant slope of the curves in this range is some indication that the fines have been substantially removed during the first two minutes of washing. Breakage of particles or reaction with the water might account for the steady decrease, but the most likely explanation is that because of the known unevenness in the weave of the No. 325 screen cloth, a sharp end point cannot be reached. The following tabulation shows the 325-mesh fineness of the four cements for washing periods of 1, 2, and 5 min. and the percentage deviation from the standard 1-min. washing period:

Cement	Percentage Passing No. 325 Sieve			Deviation from 1-min. Values, per cent	
	1 min.	2 min.	5 min.	2 min.	5 min.
Coarse—no flakes	77.7	78.7	81.0	+1.3	+4.2
Medium—small amount of flakes	89.5	90.4	91.6	+1.0	+2.3
Medium—no flakes	89.2	90.3	91.8	+1.2	+2.9
Fine—appreciable amount of flakes	93.1	93.9	95.3	+0.9	+2.4

It is evident that prolonging the washing period had the greatest effect with the coarsest cement and became less as the fineness of the cements increased. The presence of flakes appeared to have a negligible effect. Their presence in the residue in substantially the same amount regardless of duration of the washing period is indication of the absence of particle breakage.

A further source of error may arise from the use of a sieve with a large correction factor. Recent tests in which two sieves, one with a plus and one with a minus correction, were used have indicated that for cements with residues exceeding that of the standard sample, the application of a constant correction rather than a sliding correction in proportion to the residue size gives the best approximation of the true residue weight. However, it is obvious that the use of a constant correction when testing a very fine cement gives an incorrect residue weight. Therefore it is now the practice of the authors to apply a constant correction for residues exceeding that of the standard sample 114-B and a sliding correction with smaller residues. However, sieves with only nominal correction factors are now used.

If the screen is not cleaned with a weak solution of hydrochloric acid after each test, an error in the 325-mesh fineness determination may occur due to cement particles collecting in the sieve openings. The following data show the residue weights of repeated tests when no acid washing was used:

Test	Residue, g.	Error (Residue), per cent	Error in 325-Mesh Fineness, per cent
1	0.1907	0	...
2	0.1910	+0.2	...
3	0.1945	+2.0	0.5
4	0.1982	+3.9	0.9
6	0.1992	+4.5	1.1
8	0.1976	+3.6	0.9

In these tests clogging began after the second test and was nearly at a maximum at the fourth test. The maximum error in the 325-mesh fineness was 1.1 per cent. The error in the third determination, the last to be run before acid washing under the present A.S.T.M. Tentative Method C 115 – 34 T, was 0.5 per cent.

These data on various factors involved in the 325-mesh fineness test indicate the need for further study of present methods. A longer washing period may give a better measure of the actual 325-mesh fineness. Adoption of the practice of washing the residue into a beaker upon the completion of the test and immediately acid washing the screen will eliminate any error from clogging of the screen.

TABLE VI.—TESTS ON TWELVE CEMENTS PRODUCED FROM SAME CLINKER.

Sample weight 0.3 g. in all tests.

Test	$2 - \log I$		Ratio, $\frac{2 - \log I_{7.5}}{2 - \log I_{60}}$	Percentage Passing No. 325 Sieve	Log Summation	Transmittancy Constant (1 g.)	Specific Surface, sq. cm. per g.	
	60 μ	7.5 μ					Individual Transmittancy Constant	Average Transmittancy Constant[a]
No. 1	0.6596	0.4535	0.688	78.8	1.2212	735	1620	1610
	0.6556	0.4461	0.680		1.2242	734	1610	1610
No. 2	0.6696	0.4584	0.685	79.7	1.2558	723	1620	1640
	0.6737	0.4672	0.693		1.2460	730	1640	1650
No. 3	0.6840	0.4828	0.707	79.5	1.2233	741	1690	1670
	0.6696	0.4622	0.691		1.2117	748	1670	1640
No. 4	0.6925	0.4802	0.693	80.4	1.2740	719	1660	1690
	0.7033	0.4895	0.697		1.2719	721	1690	1720
No. 5	0.6990	0.4841	0.693	81.8	1.2849	726	1690	1710
	0.7011	0.4881	0.697		1.2527	744	1740	1710
No. 6	0.7055	0.4855	0.688	81.6	1.2955	718	1690	1720
	0.6968	0.4763	0.683		1.2937	720	1670	1700
No. 7	0.7825	0.5686	0.727	85.9	1.3111	746	1950	1910
	0.7696	0.5498	0.714		1.3016	752	1930	1880
No. 8	0.7905	0.5686	0.718	85.9	1.3659	717	1890	1930
	0.7986	0.5834	0.731		1.3054	750	2000	1950
No. 9	0.7100	0.4976	0.702	82.7	1.2713	741	1750	1730
	0.7235	0.5072	0.702		1.2792	737	1780	1770
No. 10	0.7305	0.5171	0.707	82.8	1.2882	733	1790	1790
	0.7328	0.5143	0.702		1.2772	738	1810	1790
No. 11	0.7258	0.4976	0.686	83.5	1.2967	735	1780	1780
	0.7235	0.5072	0.702		1.2792	744	1800	1770
No. 12	0.6861	0.4724	0.688	81.5	1.2791	727	1660	1680
	0.6990	0.4841	0.693		1.2705	732	1710	1710

[a] Average of all transmittancy constants = 733.

CHARACTER OF PRESENT RESULTS

An example of the character of the results now being obtained is shown in Table VI. All of the cements of this group were produced from a single stock of clinker during a series of efficiency tests on commercial clinker grinding mills at one plant. With one exception, duplicate tests on each cement agreed quite closely. Cement No. 8 showed a 5.6 per cent spread in specific surface when individual transmittancy constants were applied, but only 1.0 per cent when the average of all transmittancy constants was used. The average transmittancy constant was 733. The largest deviation from the average was 2.6 per cent and the average deviation 1.2 per cent. The maximum difference in specific surface when using the average transmittancy constant was 40 sq. cm. or 2.3 per cent. Six of the tests show differences of 1 per cent or less.

The material improvement in the performance of the turbidimeter has been accomplished chiefly through the adoption of a more rigid testing technique. Using present methods, there is a reasonable certainty of reproducing results within about ± 1 per cent of the average. Occasionally higher differences occur, but these can be easily detected through failure to obtain close agreement in the expression $2 - \log I_{60}$ and in the transmittancy constants.

CONCLUSIONS

The data presented indicate that inability to obtain a high degree of concordance among repeated turbidimeter tests is mainly the result of failure to establish sufficiently rigid technique and uniform conditions of test. With careful attention to these, the Wagner turbidimeter appears capable of giving results reproducible within about ± 1 per cent of the average.

The following are the more important conclusions from these studies:

1. While the use of the present testing procedure may not supply an absolute measure of specific surface of a cement, it is believed from the data now available that this instrument gives as reliable a measure of the surface as any method known today.
2. The brush used in the dispersing operation should be operated at a constant speed of about 3500 r.p.m.
3. With the dispersing procedure used in these tests, varying the test-tube diameter from 0.84 to 0.94 in. when using a stirring brush 0.75 in. in diameter did not affect the results.
4. If any new surface is produced in the brushing operation when testing cements it is of little significance.
5. An appreciable amount of new surface is produced in the brushing operation when testing samples from the impact grindability test containing large percentages of plus 200-mesh material. An adequate correction for the new surface can be obtained from a supplementary test on the plus 200-mesh material.
6. The kerosine should not be re-used, even though carefully cleaned by activated carbon followed by filtration.
7. Reliable results can be secured from samples which have been carefully stored in sealed containers for long periods of time.
8. The location of the instrument should be such that variation between the temperature of the kerosine and the air is small.
9. The settling tank must be thoroughly cleaned by mechanical means after each test.
10. Washing the No. 325 screen with a 10 per cent solution of hydrochloric acid after each fineness determination is advisable.
11. Wherever possible, an average transmittancy constant rather than the individual constant should be used in calculating the specific surface.

Further studies seem necessary on the following important features of the test: (1) dispersing operation, (2) 325-mesh fineness determination, and (3) standardization. A proposed method of standardization is described in this paper. However, conclusions on only certain features of the present dispersing method and the 325-mesh fineness determination have been reached from the data presented, leaving many important questions still unanswered.

Acknowledgments:

Many of the studies and methods of interpretation of this paper were suggested by J. Homer Griffith, Assistant Chief Chemist, Bonner Springs plant, Lone Star Cement Corp. L. A. Wagner, Research Chemist, Missouri Portland Cement Co., inventor of the turbidimeter, also offered valuable suggestions and carefully reviewed the manuscript.

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(1) L. A. Wagner, "A Rapid Method for the Determination of the Specific Surface of Portland Cement," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 553 (1933).

(2) Raymond Wilson, "A Method of Estimating the Efficiency of Pulverizers," *Mining Technology*, May, 1937, Vol. 1, No. 3.

(3) Unpublished work by the Manufacturing Research Bureau of the Portland Cement Assn.

(4) J. C. Pearson and W. W. Sligh, "An Air Analyzer for Determination of the Fineness of Cement," *Technologic Paper 48*, Nat. Bureau Standards (1915).

DISCUSSION

Mr. Edward C. Reid[1] (*presented in written form*).—In many instances the findings of this laboratory are in agreement with those set forth in the paper by Messrs. Ponzer and MacPherson. This is particularly true in the following specific cases:

1. The development of the proper technique is necessary in order to make a valuable and accurate instrument of the turbidimeter.

2. Proper standardization should include adjustment of the light intensity to 100 microamperes.

3. The use of a standard sample for calibration has numerous disadvantages.

4. There is good and sufficient reason for belief that the Standard Sample is not uniform.

5. The apparatus should be located in an air-conditioned room.

6. No appreciable new surface is produced during stirring.

7. The assumption made by Wagner for the average diameter of particles less than 7.5 μ in diameter undoubtedly introduces appreciable differences in the fineness results as compared with results from microscopic study.

8. The best procedure appears to require the use of a test tube of the same internal diameter as that of the stirring brush.

9. The kerosine should not be re-used.

10. The timing burette undoubtedly introduces appreciable errors unless the turbidimeter is located in an air-conditioned room.

11. The chief cause of error due to temperature change is the setting up of convection currents in the suspension.

12. Although the procedure has not been followed in this laboratory, the reported technique for emptying and re-using the test tube seems advisable as a precaution against losses of fine particles.

13. The conclusions drawn are in good agreement with the conclusions of this laboratory except for the following:

(*a*) A stirring speed of 3500 r.p.m. will cause the formation of a vortex in the test tube and consequently whip air into the suspension. Any such air included will have a noticeable effect on the fineness because it will attach itself to the particles and lower their speed of settlement.

(*b*) It is rather difficult to believe that there can be any production of new surface from particles larger than 200 mesh because these must by necessity be the hardest particles of any cement and since they have not been affected by the action of mill grinding, they surely will not be affected by any such minor grinding action as that of the stirring procedure. It seems more reasonable to believe that the decrease in the initial reading and the apparent increase in surface are due to air whipped into the suspension by the stirring and oscillation. As the speed of stir or the time of stir increased, the quantity of air included would also increase. This conclusion appears to be based on false evidence.

(*c*) Since it has been observed in this laboratory that CO_2 and moisture have a definite and lasting action on cements there would seem to be little ground for the statement that reliable results can be achieved with samples stored for long periods of time, even though they are carefully sealed. It has been our experience that this may be true but it is not necessarily so. Such results seem to be dependent on the extent of exposure to CO_2 and moisture prior to sealing.

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(*d*) Mechanical cleaning of the settling tank has been found necessary only occasionally, provided sufficient oleic acid is used. As much as 14 drops has been found advisable with very fine cements without the fineness showing appreciable change.

(*e*) Careful checks in this laboratory have shown that greater error in the sieve residue is introduced by frequent washing with hydrochloric acid than by clogging of the sieve openings with continued use. This is true up to 15 successive tests provided the sieve is properly cleaned each time.

(*f*) It has been found inadvisable to use an average transmittancy constant since there appears to be insufficient uniformity in mill grinding. Bad errors were introduced in almost every instance when comparisons were made between complete individual runs and runs based on an average transmittancy constant or a "single shot" method.

There seem to be several discrepancies and possible sources of error implied or reported regarding the procedure used for these determinations. They are as follows:

1. Apparently "oscillation" of the settling tank was used as a means of final dispersion of the suspension. If this was the case the results are undoubtedly inaccurate because "oscillation" whips air into the suspension that has a direct effect on the settling rate of the particles, particularly the finer portions. A manually operated plunger appears to be preferable for the final dispersion of the sample.

2. If the microammeter is tapped with a pencil prior to each reading it appears to be accurate within 0.1 microampere.

3. The use of a fixed transmittancy constant does not appear to be advisable, for particle color, shape, and opacity are not at all uniform in the same bin, since one bin usually represents more than one "grind."

4. Tests in this laboratory have shown that the No. 325 sieve can be so handled that one minute of washing will give an end point (within practical limits).

5. The suggested procedure of washing the sieve residues into a beaker rather than drying them in the sieve seems to offer another possibility of error through loss by dusting and spilling. In any event it is an added operation and does not seem necessary if the proper procedure is followed when the dried sieve is emptied.

6. The suggested method of standardization seems to be a good one with one exception, namely, it fails to compensate for the wide range in color of cements from different mills. It would seem more fair to the individual mill and just as accurate to set the initial light intensity at 100 microamperes with the cement to be tested. With any method such as that suggested or such as the use of a Standard Sample, those mills whose product is light in color are handicapped while mills producing a dark product are appreciably benefited.

7. No tendency toward stratification of the sample during stirring has been observed in this laboratory unless a vortex was formed in the test tube or the stirring speed was below 900 r.p.m.; hence it would seem unnecessary to

slow production down by manipulating the test tube manually.

8. It would seem that such reported results as are dependent on the ratio of 2-log $I_{7.5}$ to 2-log I_{60} are of doubtful accuracy since the first and last readings and the range are not necessarily measures of the specific surface any more than is the No. 325 sieve. In fact dependence on such a ratio can lead to very appreciable errors.

In general the paper is surely aimed at the right goal and can be used as a sound basis in many respects from which to work in the attempt to achieve a standardized and rigid technique for the use of the Wagner turbidimeter.

MR. P. S. ROLLER[2].—Although this paper concerns itself with details of measurement with the Wagner turbidimeter, I should like to call attention to a more general and more important matter that engaged my attention some time ago during analysis of results obtained with the instrument.

It is recognized that the figure for surface area is not correct in an absolute sense, due to irregularities of shape and surface of the particles. However, it is commonly and naturally taken for granted that the surface area is correct in a relative sense. This, however, is not true as regards the Wagner calculation for surface area.

If one compares two results obtained with the turbidimeter, for example 5000 and 2500 sq. cm. per g., one will naturally assume that, regardless of the true value of the surface area, the one material is relatively twice as fine as the other. Actually, however, it will be more than twice as fine, and by an indeterminate amount, perhaps 3, 5, or 10 times, or more.

The reason for this defective result is a serious limitation of the Wagner equation for determining surface area.[3] This consists in the fact that regardless of the fineness of the material being tested, S will not calculate to be greater than 5080 sq. cm. per g., as is easily proved by substituting the limiting value $I_{7.5}$ for all the other I's in the equation for S. This imperfection in the equation makes itself felt all the way down the line, although to a lesser extent the smaller the surface area.

In the case of portland cement, typical results for surface area are: For normal cement, Wagner 1620, "correct" 1840, difference 220; for high-early-strength cement, Wagner 2830, "correct" 4080, difference 1250. It is observed that the difference is appreciable and becomes quite large as S approaches the limiting value of 5080. From a mill standpoint, a practical objection to the defect is that when a mill grinds extra units of surface area, much more area is actually being produced than appears from the Wagner result, and an indeterminate amount of surplus grinding is therefore being done.

The result labeled "correct" in the above comparison was deduced by application of a distribution function.[4] This result is impaired in this special case by the fact that a deviation exists, which is due most likely to extraneous effects of color, etc., that are present in the turbidimeter method of analysis. However the deviation is small compared to the differences noted. It is hoped to report in somewhat greater detail later on this subject.

MESSRS. H. S. PONZER[5] AND D. R. MACPHERSON[5] (*authors' closure by letter*).—The discussion offered by Mr. Reid is based upon the experiences gained from a great amount of work with the turbidimeter during the past few years, and the authors are therefore pleased to note the many points on which he agrees. Of the more important points in question, the following comments are offered.

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[2] Associate Physical Chemist, U. S. Bureau of Mines, College Park, Md.

[3] L. A. Wagner, "A Rapid Method for the Determination of the Specific Surface of Portland Cement," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 553 (1933).

[4] P. S. Roller, "Law of Size Distribution and Statistical Description of Particulate Materials," *Journal*, Franklin Inst., Vol. 223, p. 609 (1937).

[5] Manufacturing Research Engineer, and Assistant Engineer, respectively, Manufacturing Research Bureau, Portland Cement Assn., Chicago, Ill.

Inclusion of air in the suspension by too rapid agitation in the stirring operation may cause a retardation of settlement of the particles. However, the experience of the authors in comparative specific surface determinations when testing an experimental stirring device which introduced a much greater amount of air than the regularly employed method, indicated that any error from this source was relatively slight when testing cements of normal fineness. The specific surfaces when using the experimental dispersing mechanism were 20 to 30 sq. cm. per g. higher in this range than when using the regular stirring device, although at higher cement finenesses the difference was a little greater. Apparently this feature of the testing technique, together with the possibility of the introduction of air during oscillation, deserves further study, but it is not believed that a serious condition of error will be found.

The evaluation of the amount of new surface produced during the brushing operation was one of the most difficult problems encountered in the development of a testing technique for determining the grindability of cement clinker by the impact grindability testing device. An elaborate series of tests indicated that a fairly large amount of new surface was created and that it was produced by abrasion of the plus 200-mesh material. A simple experiment involving the brushing of a coarse fraction at moderate speeds will demonstrate that it is relatively easy to create an appreciable amount of new surface. Subsequent tests of intermediate products taken during mill efficiency tests have also indicated clearly that whenever appreciable quantities of coarse material are present new surface is created in the brushing operation.

The use of an average transmittancy constant is not always advisable, as Mr. Reid has brought out. However, in many instances this procedure is highly desirable not only to bring comparable tests into better agreement, but to aid in detecting tests which are grossly in error. Obviously the use of this method depends upon an intimate knowledge of the history of the clinker employed in producing the cement under test.

Mr. Roller has pointed out a weakness of the turbidimeter calculation which casts some doubt on the reliability of results obtained with the instrument. The data submitted here and in his published paper[4] are of interest to all in the cement industry, and particularly to those engaged in research. It would appear that if the specific surface values which Mr. Roller gives as "correct" are the proper values, much of the so-called inefficient grinding when producing high-early-strength cements is truly efficient grinding, and that in many instances grinding efficiency actually increases rather than decreases as the fineness of the product is raised. This appears to be contrary to the generally accepted theories of ball and tube milling as affected by the ratio of ball to particle size.

Further work by Mr. Roller or others in critically examining the reliability of devices for measuring the specific surface of cements undoubtedly will lead to valuable discoveries of a fundamental nature. However, it should be mentioned that experiences from many sources have indicated a fairly direct and linear relationship between specific surface as measured by the Wagner turbidimeter and the strength developed by cement.

MEASURING YOUNG'S MODULUS OF ELASTICITY BY MEANS OF SONIC VIBRATIONS

By T. C. Powers[1]

Synopsis

A dynamic method of measuring Young's modulus of elasticity of a concrete or mortar specimen by determining its natural frequency of vibration is described. Data are presented comparing the moduli of elasticity obtained by this method with those obtained by static loading methods. The dynamic method is believed to give the true elastic modulus, not complicated by plastic flow.

In any complete textbook on sound[2] there may be found the equivalent of the following expression:

$$N = \frac{m^2 k}{2\pi L^2}\sqrt{\frac{E}{d}} \quad \ldots\ldots(1)$$

This equation states that the frequency of vibration N of a bar or rod of elastic material vibrating without restraint is directly proportional to the square root of Young's modulus E, inversely proportional to the square root of the density d of the material, inversely proportional to the square of the length L of the bar, and directly proportional to the radius of gyration k. L, d, and k can be measured directly, and m, a constant depending primarily on the mode of vibration, can be evaluated, as will be discussed later. Then, with E known, N can be calculated, or with N known, E can be found.

It is with respect to this latter possibility, the determination of Young's modulus by measuring the natural frequency of vibration, that this paper is concerned. Experiments have been completed which show the feasibility of calculating E of concrete and mortar from measurements of N without recourse to laborious stress-strain measurements. The values obtained are fully as reliable for some purposes as those obtained in the usual manner.

This method has been applied to such building materials as tile, slate and brick by Grime[3] and to lead, ebonite wall board, paraffin wax, rock asphalt, and asphalt mastic by Grime and Eaton.[4] Those investigators measured N by attaching a small piezo-electric quartz crystal to the specimen, striking the specimen with a light hammer, picking up and amplifying the alternating voltage thus set up and recording it on a photographic plate with a cathode-ray oscillograph. Tykociner and Woodruff[5] worked with quartz bars, measuring N electrically. Davies and Thomas[6] used a vibration method to determine Young's modulus of bars of imperfectly elastic nickel and nickel alloys. The natural frequency of vibration in this work was determined by fixing one end of the test piece and attaching to the other a magnetic armature (stalloy) placed in an alternating magnetic field. The frequency at resonance with the test bar was observed. After suitable corrections this gave the required value of N.

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[1] Assistant to Director of Research, Portland Cement Assn., Chicago, Ill.

[2] A. B. Wood, "A Text-Book of Sound," The MacMillan Co., New York City (1930); E. H. Barton, "Text-Book of Sound," The MacMillan Co., New York City (1908).

[3] G. Grime, "The Determination of Young's Modulus for Building Materials by a Vibration Method," *Philosophical Magazine*, Vol. 20, pp. 304–310 (1935).

[4] G. Grime and J. E. Eaton, "The Determination of Young's Modulus by Flexural Vibrations" *Philosophical Magazine*, Vol. 23, pp. 96–99 (1937).

[5] J. T. Tykociner and M. W. Woodruff, "Flexural Vibrations of Piezo-Electric Quartz Bars and Plates," *Bulletin 291*, Engineering Experiment Station, University of Illinois (1937).

[6] R. M. Davies and I. H. Thomas, "A Study of an Electrically Maintained Vibrating Reed and Its Application to the Determination of Young's Modulus," *Philosophical Magazine*, Vol. 23, pp. 361–396 (1937).

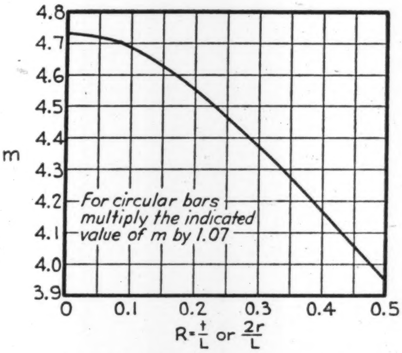

FIG. 1.—Relation Between m and Ratio of Depth to Length, R, for a Rectangular Bar Vibrating Laterally in the Fundamental Mode.

Equation of line:

$$1 - \cos\left\{m\left[\left(1+\frac{m^4R^4}{24^2}\right)+\frac{m^2R^2}{24}\right]^{\frac{1}{2}}\right\}$$
$$\cosh\left\{m\left[\left(1+\frac{m^4R^4}{24^2}\right)^{\frac{1}{2}}-\frac{m^2R^2}{24}\right]^{\frac{1}{2}}\right\}$$
$$+\left(\frac{3m^2R^2}{24}+\frac{4m^6R^6}{24^3}\right)\sin$$
$$\left\{m\left[\left(1+\frac{m^4R^4}{24^2}\right)^{\frac{1}{2}}+\frac{m^2R^2}{24}\right]^{\frac{1}{2}}\right\}$$
$$\sinh\left\{m\left[\left(1+\frac{m^4R^4}{24^2}\right)^{\frac{1}{2}}-\frac{m^2R^2}{24}\right]^{\frac{1}{2}}\right\}=0$$

After W. P. Mason, "Motion of a Bar Vibrating in Flexure, Including the Effect of Rotatory and Lateral Inertia," *Journal*, Acoustical Soc. America, Vol. 6, p. 246 (1935.)

In the work done by the author, reported below, advantage was taken of the fact that N may be measured by matching, with any suitable calibrated source of tone, the pitch of the musical tone given by the specimen when struck with a small hammer. A sonometer is perhaps the best instrument for this purpose, though a vacuum tube oscillator may also be suitable. For the work reported below, the frequency was determined by finding the bar in a set of orchestra bells[7] which gave the pitch nearest to that of the specimen. The maximum uncertainty in determining frequency by this method is one-fourth tone, or about 3 per cent of the frequency; the actual uncertainty was usually about one-eighth tone, or roughly 1½ per cent. More recently a home-made sonometer has been adopted for routine measurements. With this instrument, which can be tuned to unison with the test specimen, the uncertainty in determining frequency is very small, for the normal ear can detect extremely small differences in pitch.

Any concrete or mortar specimen can be tested if it is symmetrical and of such dimensions that its natural vibration frequency falls in a suitable range. Most of the specimens used thus far have been 2 by 2 by 9½ in. (5.08 by 5.08 by 24 cm.); mortar prisms which were made for volume change tests. These were found suitable except for very rigid mortars giving values of N over 3000. For such mortars a smaller ratio of thickness to length would be better. Miscellaneous larger specimens have been used also.

Relationship Between E and N:

Before giving more details of the method, the basis for calculation of E must be considered further. When a bar vibrates, a cross-sectional element may be thought of as executing two movements: a motion of translation laterally, and one of rotation relative to the position of the unbent neutral axis. In the derivation of Eq. 1 the rotatory inertia was neglected for the sake of simplicity and because in most cases the error introduced thereby is not large. But if the thickness of the bar is a relatively large fraction of the length, as it is in most concrete specimens, the rotatory inertia must be taken into account.

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[7] "Orchestra bells" are sets of tuned steel bars.

Lord Rayleigh[8] gives an approximate correction and Tykociner and Woodruff give others, including a semi-empirical equation to fit their particular data. The most direct and exact corrections are given by Mason[9] in terms of *m* as affected by the ratio of thickness to length. The equation for this correction, and its graphical representation, for the fundamental mode of vibration is given in Fig. 1. If m' represents the value of m corresponding to the given ratio of thickness to length, then Eq. 1 may be made applicable to the present purpose by writing it as follows:

$$E = \frac{4\pi^2 N^2 dL^4}{k^2(m')^4} \quad \text{........(2)}$$

As a specific example, Eq. 2 may be solved for a 2 by 2 by 9½ in. (5.08 by 5.08 by 24 cm.) bar, which happens to be the size to which much of the data reported below pertain.

The values to be substituted are:

$$L^4 = 24^4 = 331{,}776$$

$$k^2 = \frac{t^2}{12} = \frac{5.08^2}{12} = 2.15$$

$$t = \text{thickness of the bar.}$$

For circular bars $k^2 = \frac{r}{4}$, r being the radius.

$$R = \frac{t}{L} = 0.21$$

whence, from Fig. 1

$$m' = 4.54$$

$$(m')^4 = 424.8$$

$$\frac{4L^4\pi^2}{k^2(m')^4} = 1.434 \times 10^4$$

$$E = N^2d\ 1.434 \times 10^4 \text{ dynes per sq. cm.} \quad (3)$$

Multiplying by the conversion factor (1.45×10^{-5}) gives

$$E = 0.208\ N^2d \text{ lb. per sq. in...}(4)$$

Equations 2 and 4 include a correction for rotatory inertia as described above, but they neglect the effect of another inertia, namely, that due to lateral contraction and expansion of the bar. This is proportional to Poisson's ratio, λ, which, for concrete, may be taken as 0.16. Mason shows that the correction for both rotatory and lateral inertia can be made in one operation with the aid of Fig. 1 simply by using instead of the ratio of thickness to length, R, a value $R\ (1 + \lambda)^{\frac{1}{2}}$.

Thus for the particular example given above,

$$R\ (1 + \lambda)^{\frac{1}{2}} = 0.21 \times 1.16^{\frac{1}{2}} = 0.227$$

and the corresponding value of m, which may be designated m'', is, from Fig. 1, 4.51.

With the notation just indicated, Equation 1 becomes, when corrected for both lateral and rotatory inertia,

$$E = \frac{4\pi^2 N^2 dL^4}{k^2(m'')^4} \quad \text{........(5)}$$

and, when the values for the 2 by 2 by 9½ in. bar are substituted,

$$E = 0.214\ N^2d \text{ lb. per sq. in....}(6)$$

Methods and Results of Tests:

The first step in the sonic method is to determine the mode of vibration of the bar. This is very easily accom-

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[8] John W. Rayleigh, "The Theory of Sound," (2 vols.) Second Edition, The MacMillan Co., London (1926).

[9] W. P. Mason, "Motion of a Bar Vibrating in Flexure, Including the Effect of Rotatory and Lateral Inertia," *Journal*, Acoustical Soc. America, Vol. 6, p. 246 (1935).

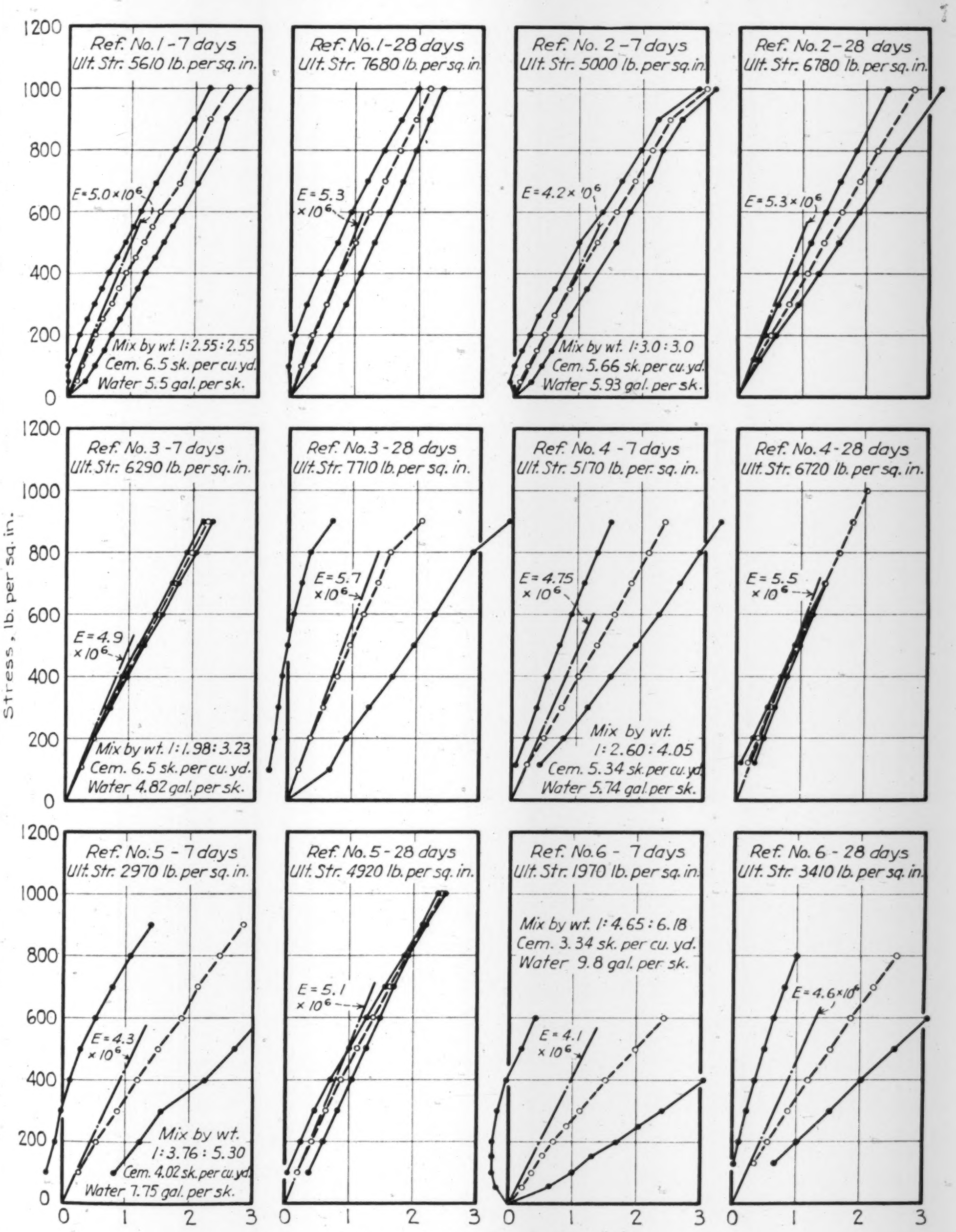

FIG. 2.—Comparison of E as Determined by the Sonic Method on 5 by 5 by 18-in. Bars with Stress-Strain Measurements Made on Companion 6 by 12-in. Cylinders Tested in Compression.

The slopes of the straight dash-dot lines are as indicated by the sonic test; the two series of solid points indicate the deflections as measured simultaneously on opposite sides of the specimen; the circles represent the average deflection. Stress-strain values represent the first loading of the specimen.

plished, if it is a small specimen, by holding the bar up by one end and then tapping along the length of the bar, noting the positions of the "dead" spots—the nodes. If the fundamental is being given, two nodes will be found, each exactly $0.224L$ from the nearest end, regardless of the dimensions of the bar. If it is the first overtone there will be three nodes, one at the middle and one $0.132L$ from either end. More than three nodes are possible, but are not likely to be detected in concrete specimens, due to their necessary thickness.

The foregoing procedure is not essential, but it aids in becoming familiar with the phenomena involved. For determining E, the bar is preferably excited in such a way as to produce the fundamental vibration; in fact this must be done if Eq. 2 or Eq. 5 is to be applied. To elicit the fundamental vibration, the bar is rested on narrow rubber or wooden supports placed at the two nodes ($0.224L$ from each end) and is struck with a light hammer at the center or extreme end. Light specimens may be held by hand as described above and struck at the middle or extreme end. Having produced the fundamental tone, its pitch may be determined by one of the methods mentioned above. When the specimens are very young, it takes a practiced ear to recognize the heavily damped musical tone through the noise of the hammer's impact, but in older specimens there is little difficulty, and with dry ones it is especially easy. As mentioned before, it is best to avoid specimens of such dimensions that the frequency is above about 3000.

For illustration, the method was used on three bars of cold-rolled steel. N was measured by means of the musical pitch of the fundamental vibration, and E was calculated from Eq. 5. The results follow:

DIMENSIONS, IN.	N, VIBRATION PER SEC.	E, LB. PER SQ. IN.
$1.0 \times 0.67 \times 10.0$	1030	29 530 000
$1.0 \times 0.67 \times 7.0$	2090	29 730 000
$1.0 \times 0.67 \times 4.0$	6200[a]	28 940 000

[a] Pitch too high to be determined with certainty.

The value of E for this particular lot of steel as determined by the usual static method is not available, but the data served to show that the sonic method gives reasonable results.

To test the practicability of the method for concrete, four mixes using Elgin sand and gravel and laboratory cement[10] were made, ranging from rich to very lean.[11] From each batch, 5 by 5 by 18-in. bars and 6 by 12-in. cylinders were made and cured in the moist room. The cylinders were used for strength tests and stress-strain measurements at 7 and 28 days. The bars were tested periodically for Young's modulus by the sonic method.

TABLE I.—YOUNG'S MODULUS OF 5 BY 5 BY 18-IN. BARS AS DETERMINED BY THE SONIC METHOD.

Series	Reference Number	Age, days	d	N	$N^2 \times 10^{-6}$	$N^2d \times 10^{-6}$	$E = 0.525N^2d \times 10^{-6}$
J294....	1	7	2.43	1975	3.90	9.48	5.0
	1	28	2.44	2040	4.16	10.15	5.3
	2	7	2.42	1825	3.32	8.03	4.2
	2	28	2.43	2035	4.14	10.06	5.3
	3	7	2.47	1950	3.80	9.39	4.9
	3	28	2.48	2100	4.40	10.91	5.7
	4	7	2.48	1910	3.65	9.05	4.75
	4	28	2.49	2050	4.20	10.46	5.5
	5	7	2.44	1830	3.35	8.17	4.3
	5	28	2.45	1990	3.96	9.70	5.1
	6	7	2.44	1800	3.24	7.90	4.1
	6	28	2.45	1900	3.61	8.84	4.6

The results of these tests are given in Table I and in Fig. 2. On each of the diagrams the plotted points show the relationship between stress and strain for the 6 by 12-in. cylinders. The strains were measured with a rotating-mirror extensometer. This instrument was provided with two mirrors which indicated the deformation on opposite sides of the specimen. The deformations of each side are plotted separately, and the average of the two is indicated also. The straight lines are drawn so as to represent the value of Young's modulus as determined by the sonic method (Eq. 5) on the 5 by 5 by 18-in. bars. It will be seen that in most cases the calculated line (heavy solid line) either follows exactly the points of the average stress-strain curve at low stresses, or else appears to be tangent to it at the origin. There is one notable exception as shown in the upper right-hand diagram of Fig. 3, where the calculated slope seems much too high for the experimental points. It should be noted that in this diagram the plotted points indicate a lower modulus of elasticity than the corresponding points for the same mix tested at 7 days (adjacent diagram) despite the fact that the compressive strength has increased. This is contrary to the general rule, and it is believed, therefore, that for some unknown reason the 28-day stress-strain values are incorrect.

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[10] A mixture of four brands purchased in Chicago.

[11] Series J-294.

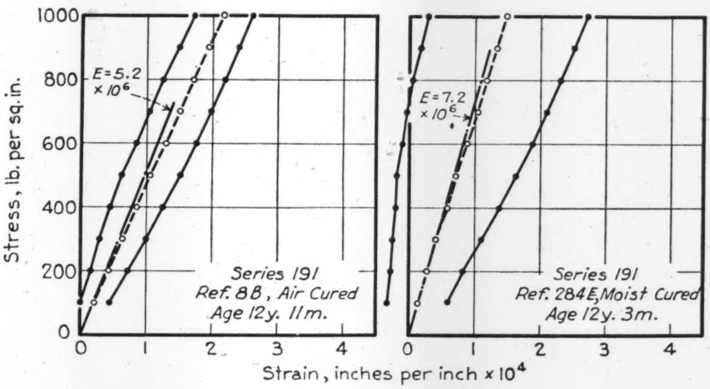

FIG 3.—Comparison of E as Determined by the Sonic Method on 7 by 10 by 38-in. Bars with Stress-Strain Measurements Made on Companion 6 by 12-in. Cylinders Tested in Compression.

The slopes of the straight dash-dot lines are as indicated by the sonic test; the two series of solid points indicate the deflections as measured simultaneously on opposite sides of the specimen; the circles represent the average deflection. Stress-strain values represent the first loading of the specimen.

Other discrepancies are found in the bottom row of diagrams in Fig. 2. In this case it should be noted that there are very large differences in the deflections of opposite sides of the test cylinders, due apparently to eccentric loading. It is doubtful that the average of readings differing this much gives the true strain for the indicated stress. The heavily loaded side would yield too much, due to plastic flow, thus giving an average curve displaced too far to the right. It is possible, on the other hand, that in these lean mixes or in richer mixes at early ages, the true stress-strain curve is bent strongly even at relatively low stress, so that the slope indicated by the sonic method, employing an almost infinitesimal strain, should coincide with but very little of the stress-strain curve.

Supplementing the tests just discussed, a number of specimens already on hand from other experimental projects were tested. Table II and Fig. 3 show the results, in the same manner as Fig. 2, of stress-strain measurements on 6 by 12-in. cylinders and of sonic tests on companion 7 by 10 by 38-in. bars. Here, where the concrete is very old (relatively), the two methods agree well within the reproducibility of the stress-strain test. The eccentricity of loading of reference No. 284E was rather bad, but with this kind of concrete the average seems to indicate the strain correctly, due, no doubt, to the fact that plastic flow was almost negligible during the loading period.

In Table III are shown results of tests on 2 by 2 by $9\frac{1}{2}$-in. bars. In these tests a center load over an 8-in. span was applied and the deflection measured by means of a Federal dial which registered changes of 10^{-4} in., permitting estimates of 10^{-5} in. These deflection measurements were made on the same specimens used for the sonic test. The value of E was calculated from the equation

TABLE II.—YOUNG'S MODULUS OF 7 BY 10 BY 38-IN. BARS AS DETERMINED BY THE SONIC METHOD.

Series	Reference Number	Age, years	d	N	$N^2 \times 10^{-6}$	$2.49^a\ N^2d \times 10^{-6}$	$4.40^a\ N^2d \times 10^{-6}$	$N^2d \times 10^{-6}$
191....	8B	13	2.40	930	0.865	2.076	5.2	...
191....	284E	$12\frac{1}{4}$	2.48	820	0.672	1.666	...	7.2

[a] For beam 8B, t = 10 in.; for beam 284E, t = 7 in.

$$E = \frac{1}{48}\frac{PL^3}{yI}$$

where P = the applied load,
L = distance between supports,
y = deflection, and
I = moment of inertia.

The specimens were of various ages and had been stored for some time in air at 50 per cent relative humidity. It will be noted that there are no discrepancies between the two methods that exceed the normal variations of the test. In this connection attention should be called to the fact that deflection measurements were anything but precise; the deflections which could be produced without danger of breaking the specimens amount to only three or four divisions on the dial. Although the results were fairly reproducible, the uncertainty in these values amounts to as much as 0.3 × 10^6, and at high values of E, somewhat more. The uncertainty of the sonic method amounts usually to about half of this value, due to the necessity of interpolating between the successive bells, as mentioned earlier in this report.

TABLE III.—COMPARISON OF RESULTS OF TWO METHODS OF DETERMINING YOUNG'S MODULUS OF ELASTICITY.

2 by 2 by $9\frac{1}{2}$-in. bars—dry.
$E = 0.214\ N^2d$.

Series	Reference Number	Mix by Weight	d	N	$N^2\ (\times 10^{-6})$	$dN^2\ (\times 10^{-6})$	Young's Modulus, lb. per sq. in. ($\times 10^{-6}$) Son Method	Young's Modulus, lb. per sq. in. ($\times 10^{-6}$) Deflection Method
267.......	27B	1:2.85	2.19	3000	9.00	19.71	4.2	3.7
	28B	1:2.85	2.22	3000	9.00	19.98	4.3	3.6
	29B5	1:3.8	2.13	2700	7.29	15.53	3.3	3.2
	30B	1:3.8	2.15	2720	7.40	15.91	3.4	3.1
	31B	1:5.2	2.03	2300	5.29	10.74	2.3	2.3
	32B	1:5.2	2.08	2400	5.76	11.98	2.6	2.5
	33A	1:8.4	1.92	1840	3.38	6.49	1.4	ND[a]
	34A	1:8.4	1.93	1840	3.38	6.52	1.4	ND[a]
	26A	1:7.1	2.13	2500	6.25	13.31	2.8	2.4
	26B	1:7.1	2.13	2580	6.66	14.18	3.0	2.5
233V....	1A	1:3.85	2.27	2870	8.24	18.70	4.0	3.5
	2A	1:3.05	2.29	3120	9.73	22.28	4.8	4.3
	3A	1:2.47	2.30	.....	.....	.....	...	4.7
	4A	1:3.80	2.25	2870	8.24	18.54	4.0	3.6
J264.....	1A - 1	1:2.0	2.24	3200	10.24	22.94	4.9	4.47
	2A - 1	1:2.0	2.20	2800	7.84	17.25	3.7	3.4
Average[b]							3.6	3.4

[a] ND = not determined.
[b] 33A and 34A omitted.

Similar data are shown in Table IV. Here, the specimens were those which had been stored in water or in the moist room and were consequently saturated with water at the time of test. In this group, calculations are shown for two different loads.

Discussion:

It was pointed out by Davies and Thomas that a dynamic method such as the one described in this paper is the only kind of measurement that can give the true elastic modulus of that class of materials which exhibits permanent set and plastic flow or time-yield or creep, as it is variously called. The usefulness of the dynamic method (whether using the sonic or some other method of measure) would seem to be confined, therefore, to whatever use can be made of a knowledge of the true elasticity of a material. An accurate value of Young's modulus could hardly be recommended as a basis for computing deflections of a concrete member, except where instantaneous values were required, because of the well-known phenomenon of plastic flow. However, it seems that in laboratory studies of the stress-strain relationships it should provide a valuable reference point from which to compute the non- or semi-elastic deformations.

One of the best features of the sonic method is that it does not destroy the test specimen nor alter it by the effect of high stress; the same specimen may be used for test at all ages. Another advantage is its simplicity, accuracy, and speed. A record of this fundamental property of a wide variety of test specimens can be obtained with very little outlay of time, or money for equipment.

TABLE IV.—COMPARISON OF RESULTS OF TWO METHODS OF DETERMINING YOUNG'S MODULUS OF ELASTICITY.

2 by 2 by 9½-in. bars—saturated.
$E = 0.214\ N^2d$.

Series	Reference Number	Age, days	d	N	$N^2 \times 10^{-6}$	$N^2d \times 10^{-6}$	Young's Modulus, lb. per sq. in. ($\times 10^{-6}$)			
							Sonic Method	Deflection Method[a]		
								A	B	Average
J294......	3A	17	2.249	2790	7.78	17.50	3.7	4.5	3.2	3.8
	3B	17	2.246	2790	7.78	17.50	3.7	3.7	3.4	3.6
	4A	17	2.298	2900	8.41	19.33	4.1	4.3	4.0	4.2
	4B	17	2.297	2900	8.41	19.33	4.1	3.8	3.7	3.8
	5A	17	2.330	3135	9.82	22.88	5.0	5.0	...	...
	5B	17	2.327	3135	9.82	22.88	5.0	5.2	4.5	4.8
	6A	17	2.335	3135	9.82	22.93	4.9	5.2	4.2	4.7
	6B	17	2.336	3135	9.82	22.93	4.9	5.0	4.3	4.8
233......	389A	13	2.277	2850	8.12	18.49	4.0	4.0	3.4	3.7
	389B	12	2.277	2790	7.78	17.72	3.8	3.7	3.5	3.6
	389C	11	2.277	2790	7.78	17.72	3.8	3.7	3.5	3.6
	390A	13	2.296	2900	8.41	19.31	4.1	4.0	3.8	3.9
	390B	12	2.296	2900	8.41	19.31	4.1	4.3	3.6	4.0
	390C	11	2.296	2900	8.41	19.31	4.1	4.3	3.7	4.0
	391A	13	2.306	3000	9.00	20.75	4.4	4.4	4.1	4.2
	391B	12	2.306	3000	9.00	20.75	4.4	5.0	4.2	4.6
	391C	11	2.306	3000	9.00	20.75	4.4	4.0	4.2	4.1
						Average[b]	4.2	4.4	3.9	4.1
J264......	1A - 2		2.36	3450	11.90	28.08	6.0	5.2	5.2	
	2A - 2		2.31	3400	11.56	26.70	5.7	5.5	4.8	
J245......	1C - 2		2.37	3510	12.32	29.20	6.2	5.2		
	2C - 2		2.33	3490	12.18	28.38	6.1	5.2		
	3C - 2		2.40	3170	10.05	24.12	5.2	4.3		
						Grand Average	4.6	4.5		

[a] Center load on 8-in span: A = load of 102 lb.; B = load of 152 lb.
[b] 5A omitted.

Incidentally, it should be possible to determine E from the velocity of sound in the concrete as well as by the dynamic method discussed in this paper, that of using flexural vibrations. Once the method were developed, it might be more generally applied than the one described in this paper.

DISCUSSION

MR. F. E. RICHART.[1]—The tests on short-time creep presented at this annual meeting by Jensen and Richart[2] and the tests reported by Jones and Richart in 1936[3] indicates that the strains as ordinarily measured in concrete may be considerably larger than if the test could be made instantaneously. Not only was the strength and modulus of elasticity greater for cylinders tested in one second than for those requiring several minutes, but we have shown that measurable creep occurs even at low loads in a period of a few seconds. This appears to be direct confirmation of Mr. Powers' observation that the values of modulus of elasticity found by sonic vibrations are slightly higher than those found by the usual extensometer test.

MR. P. J. FREEMAN.[4]—Can we apply this method in the case of a simple cylinder?

MR. T. C. POWERS.[5]—We have successfully applied this method to 6 by 12-in. cylinders in a few instances. In general, however, it is not practicable, because the ratio of thickness to length in such specimens is too large, giving a pitch too high for the ear to identify it accurately. It has been found that the length of the specimens should be at least 5 times its thickness.

MR. C. H. SCHOLER.[6]—It would seem to me that the method presented by Mr. Powers might be very advantageous in studying the durability of concrete under various conditions of exposure. It is probable that the elastic modulus of the concrete is one of the first properties affected by deteriorating influences such as freezing and thawing or the cracking of the paste around sand grains during the shrinkage of the paste in drying and cooling. I should like to inquire whether Mr. Powers has made any attempt to use this method in connection with durability studies.

MR. POWERS.—No, we have done little with this test, other than what has been reported this evening. There are several possible applications not yet explored in our laboratory.

MR. P. H. BATES.[7]—At the Bureau of Standards we are about to start an investigation from which we hope to find out what takes place during freezing and thawing, other than looking at the bar and saying "It does not look good," etc. We are adopting a so-called sonic method. However, we do happen to have the physical equipment which Mr. Powers referred to. It really does not cost so much.

MR. W. F. KELLERMANN.[8]—With further reference to Mr. Scholer's question, we used a rough approximation of the sonic method for determination of modulus of elasticity in connection with a series of freezing-and-thawing tests on disks cut from the tops of concrete cores. The disks would be held in the fingers and rapped lightly with a hammer and, although no attempt was made to measure the modulus of elasticity, we could tell when they became unsound. We found that, once the sound changed to indicate unsoundness in the specimens, it would not require many more alternations of freezing and thawing before they could be readily broken apart by hand.

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[1] Research Professor of Engineering Materials, University of Illinois, Urbana, Ill.

[2] Russell S. Jensen and Frank E. Richart, "Short-Time Creep Tests of Concrete in Compression," see p. 410.

[3] Paul G. Jones and F. E. Richart, "The Effect of Testing Speed on Strength and Elastic Properties of Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 380 (1936).

[4] Principal Materials Engineer, Tennessee Valley Authority, Knoxville, Tenn.

[5] Assistant to Director of Research, Portland Cement Assn., Chicago, Ill

[6] Professor of Applied Mechanics, Kansas State College of Agriculture, Road Materials Laboratory, Manhattan, Kans.

[7] Chief, Clay and Silicate Products Division, National Bureau of Standards, Washington, D. C.

[8] Associate Materials Engineer, U. S. Bureau of Public Roads, Washington, D. C.

MR. IRA PAUL.[9]—In my field examination of concrete pavements, the same idea of tapping the slabs with a hammer was used. When the concrete was sound it had a vibrant resonance under the impact of the hammer, while disintegrated concrete seemed to have a dead ring. I think there may be some application of this test method in conducting such surveys.

MR. J. C. PEARSON.[10]—If you should saw one of these bars out of a piece of concrete containing normal aggregates, say up to $1\frac{1}{2}$ in. in size, would you be able to determine its modulus by the same method?

MR. POWERS.—We have not tried that, but I think it would be possible to make the test on such a specimen. However, if the cross-section of the specimen were smaller than that of the larger aggregate particles, thus giving a prism made up of alternate sections of stone and mortar, the interpretation of the results might be difficult. If the specimen were large enough to be considered a true sample, the correct value for the modulus of elasticity should be obtained.

(*Author's closure*).—Subsequent to the presentation of the paper, we have used this test in connection with durability studies in the manner mentioned by Mr. Scholer and others. Concrete prisms, 3 by 3 by 15 in., were subjected to freezing and thawing, the modulus of elasticity being determined every few cycles by the sonic method. After from 3 to 20 cycles, depending upon the quality of the concrete, the value for the modulus of rupture as determined by this method of test began to drop, giving a very definite indication of damage to the specimen before there was visible indication of failure. It thus appears that this test might make it possible to rate the relative behavior of test specimens on the basis of fewer cycles than are generally found necessary when judging the results according to appearance or loss in weight.

In connection with the general application of the test, occasions may arise in which it is necessary to have a knowledge of the approximate modulus of elasticity of the material being tested. This is due to the fact that it is psychologically difficult at times to determine definitely whether two tones are identical or one or more octaves apart. For example, a specimen might give the same apparent pitch as high C, about 1056 vibrations per second,[11] while in reality its pitch might be one octave higher, 2112 vibrations per second. If such a mistake were made, the calculated modulus of elasticity would be only one-fourth its true value. Such a large mistake can be detected without difficulty, provided the general order of magnitude of the modulus of elasticity of the material being measured is known.

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[9] Associate Laboratory Engineer, New York State Department of Public Works, Division of Engineering, Albany, N. Y.

[10] Director of Research, Lehigh Portland Cement Co., Allentown, Pa.

[11] "A" = 440 vibrations per second. standard pitch.

FREEZING-AND-THAWING TESTS FOR BUILDING BRICK[1]

By J. W. McBurney[2] and A. R. Eberle[3]

Synopsis

This paper reports the effect, on the resultant disintegration of certain selected and presumably comparable samples of brick, of certain variations in the method of making freezing-and-thawing tests. Direct comparisons are made of such variables as: (1) the use of whole bricks *versus* half bricks for test specimens; (2) freezing in contact with water and freezing in air; (3) oven drying after every third cycle *versus* partial drying and absence of drying; and (4) different durations of soaking time. Other samples were subjected to the action of weather, using three types of exposure.

The action of both laboratory freezing methods and natural exposures can be summed up by stating that the greater the degree of saturation at the time of freezing, the more effective was the method in producing disintegration.

Introduction

That different methods of making a freezing-and-thawing test will produce different results on bricks of like nature was recognized as early as 1907 by Jones (1).[4] However, except for drain tile (2) there is today a total lack of standards for making freezing-and-thawing tests on structural clay products. Since both the methods of test and materials tested were variable, little information is available in the literature with respect to the independent effect of variations in methods.

A particular method of freezing and thawing (3) has been used at the National Bureau of Standards since 1930 in the study of the durability of certain of the bricks collected as part of the 1930 survey (4). A good correlation is found between the degree of disintegration resulting from the use of this particular method of freezing and thawing and that property of the brick which is measured by the ratio of the absorption resulting from 24-hr. immersion in water at room temperature to the absorption after 5-hr. boiling and cooling (5). The use of this ratio (now called C/B) therefore provides a means for selecting and sorting bricks so that the variability of the samples will be reduced.

This paper reports the effect, on the resultant disintegration of certain selected and presumably comparable samples of brick, of certain variations in the method of making freezing-and-thawing tests. Direct comparisons are made of such variables as: (*a*) the use of whole bricks *versus* half bricks for test specimens; (*b*) freezing in contact with water and freezing in air; (*c*) oven drying after every third cycle *versus* partial drying and absence of drying; and (*d*) different durations of soaking time. Other samples were subjected to the action of weather, using three types of exposure. Obviously, only a few of the many possible variations in the technique of making freezing-and-thawing tests have been included in this work.

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[1] Publication approved by the Director of the National Bureau of Standards of the U. S. Department of Commerce.

[2] Senior Technologist, National Bureau of Standards, Washington, D. C.

[3] Research Associate of the Structural Clay Products Institute at the National Bureau of Standards, Washington, D. C.

[4] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 483.

SELECTION, PREPARATION AND PROPERTIES OF THE BRICK SAMPLES

Selection of Samples:

The data secured in the 1930 Survey (4) made possible the selection of five samples which corresponded very closely to previous samples with known properties. These samples consisted of 300 brick each, purchased from five manufacturers. The descriptions of these shipments follow.

TABLE I.—DISTRIBUTION OF BRICK A BY C/B RATIO.

C/B Classification	Distribution of Bricks in Large Sample, number of specimens	Distribution of Bricks in Each Sample of 20, number of specimens
0.825	1	..
0.830	8	..
0.835	19	1
0.840	32	2
0.845	28	2
0.850	38	3
0.855	41	3
0.860	54	4
0.865	37	3
0.870	15	1
0.875	12	1
0.880	4	..
0.885	3	..
0.890	2	..
Total	294	20

Description of Types of Brick:

A is a "light hard" dry-press clay brick with a narrow range of strength, of water absorption and of C/B ratio.

B is a definitely underburned side-cut shale brick having an abnormally low strength and high water absorption for a shale brick.

C is a soft mud clay brick, rather underburned, and with a low strength and high water absorption. The principal peculiarity of the C bricks is their rather low C/B ratio extending over a considerable range.

TABLE II.—DISTRIBUTION OF SPECIMENS IN SUBSAMPLES OF BRICKS A, B, C, D and E CLASSIFIED BY C/B RATIO.

C/B Ratio Range	Number of Specimens						
	Sample A	Sample B	Sample C	Sample D	Sample E	Sample B-E	Sample A-E
0.650 to 0.670	....	....	2	....	....	2	2
0.675 to 0.690	....	....	2	....	....	2	2
0.695 to 0.710	....	....	1	....	....	1	1
0.715 to 0.730	....	....	1	....	....	1	1
0.735 to 0.750	....	....	4	....	....	4	4
0.755 to 0.770	....	....	3	....	....	3	3
0.775 to 0.790	....	....	2	....	....	2	2
0.795 to 0.810	....	3	2	....	....	5	5
0.815 to 0.830	....	8	2	1	5	16	16
0.835 to 0.850	8	8	1	1	9	19	27
0.855 to 0.870	11	1	....	4	4	9	20
0.875 to 0.890	1	....	....	10	2	12	13
0.895 to 0.900	....	....	....	4	....	4	4
Total	20	20	20	20	20	80	100

TABLE III.—AVERAGE, MAXIMUM AND MINIMUM VALUES OF C/B RATIO, WATER ABSORPTION BY 24-HR. COLD IMMERSION, AND WATER ABSORPTION BY 5-HR. BOILING FOR SUBSAMPLES 1 TO 12 OF BRICK C.

Sub-sample	C/B Ratio			Water Absorption					
				24-hr. Cold			5-hr. Boiling		
	Average	Maximum	Minimum	Average, per cent	Maximum, per cent	Minimum, per cent	Average, per cent	Maximum, per cent	Minimum, per cent
C-1	0.751	0.835	0.650	17.2	20.5	12.8	22.7	25.5	19.3
C-2	0.752	0.835	0.660	17.4	20.7	12.9	23.0	25.9	19.6
C-3	0.754	0.840	0.655	17.5	20.7	13.0	23.0	26.0	19.5
C-4	0.750	0.820	0.660	17.4	20.8	13.1	23.0	25.8	19.8
C-5	0.751	0.820	0.660	17.3	21.0	13.3	22.9	26.1	19.9
C-6	0.751	0.820	0.660	17.2	20.8	12.8	22.8	25.4	19.3
C-7	0.752	0.825	0.660	17.6	20.8	13.1	23.3	25.8	19.8
C-8	0.751	0.825	0.650	17.2	20.9	13.1	22.9	25.4	19.2
C-9	0.751	0.825	0.655	17.2	20.3	12.9	22.8	25.2	19.5
C-10	0.752	0.825	0.660	17.3	22.0	13.2	22.9	27.1	19.4
C-11	0.752	0.825	0.650	17.2	20.4	13.3	22.8	25.1	19.0
C-12	0.751	0.825	0.655	17.1	20.8	13.0	22.5	25.3	19.5

D is a side-cut clay brick having a strength above average and a medium water absorption. A high C/B ratio was the reason for including brick D in the series.

E is an end-cut clay brick classified as "light hard" by the manufacturers. The strength and water absorption is medium, as is the C/B ratio.

Table IV.—Average, Maximum and Minimum Values of C/B Ratio, Water Absorption by 24-hr. Cold Immersion and by 5-hr. Boiling, Compressive Strength and Modulus of Rupture for Subsamples 1 and 11 of Bricks A, B, C, D and E.

Description of Test	Subsample		Brick A	Brick B	Brick C	Brick D	Brick E
C/B Ratio	1	Avg.	0.855	0.829	0.751	0.878	0.845
		Max.	0.875	0.855	0.835	0.900	0.885
		Min.	0.835	0.800	0.650	0.830	0.815
	11	Avg.	0.855	0.830	0.752	0.877	0.842
		Max.	0.875	0.860	0.825	0.900	0.880
		Min.	0.835	0.800	0.650	0.840	0.815
Water absorption by 24-hr. cold immersion, per cent	1	Avg.	14.9	15.3	17.2	12.8	14.9
		Max.	16.8	16.6	20.5	15.4	16.0
		Min.	12.6	13.9	12.8	8.3	13.1
	11	Avg.	15.7	15.7	17.2	11.8	14.6
		Max.	19.6	17.4	20.4	14.6	15.8
		Min.	13.8	14.3	13.3	9.2	13.1
Water absorption by 5-hr. boiling, per cent	1	Avg.	17.5	18.5	22.7	14.5	17.6
		Max.	20.2	19.7	25.5	17.1	19.2
		Min.	14.9	17.3	19.3	9.8	16.1
	11	Avg.	18.4	18.9	22.8	13.5	17.4
		Max.	22.4	20.6	25.1	16.2	18.6
		Min.	16.5	17.8	19.0	10.7	16.1
Compressive strength, half bricks tested flatwise, lb. per sq. in.	1	Avg.	2985	1620	1880	6 815	2825
		Max.	4000	2230	3000	9 800	3460
		Min.	1625	1010	890	4 175	2235
	11	Avg.	2705	1920	1945	8 200	2990
		Max.	3700	2510	2760	11 500	3850
		Min.	1550	1330	960	4 900	2430
Modulus of rupture, lb. per sq. in.	1	Avg.	293	200	473	1 185	472
		Max.	474	300	770	2 200	704
		Min.	179	135	240	620	277
	11	Avg.	277	244	504	1 205	550
		Max.	407	407	879	1 785	755
		Min.	91	132	180	720	326

Preparation and Properties of Subsamples:

Water absorption by 24-hr. cold immersion and by 5-hr. boiling was determined on each brick (total 1500) according to the A.S.T.M. Standard Methods C 67 – 37 (**6**). From these data the C/B ratio of each specimen was calculated to the nearest 0.005. From each sample of 300 bricks, a set of 12 subsamples, consisting of 20 bricks each, was then prepared so that each subsample had, as far as the accuracy of the test method permitted, the same range and distribution of specimens with respect to C/B classification. How this was done is illustrated by the distribution data for brick A given in Table I.

The C/B distribution of brick C was such that grading to the nearest 0.005 C/B ratio did not provide a minimum of 12 specimens in each group. Brick C was therefore in part classified by 0.015 C/B differences. A similar combination of groupings was used on the upper and lower grading of Bricks D and E. There is evidence that the C/B ratio is not reproducible to better than ±0.0075 (**7**). Table II shows the composition according to C/B ratio of the subsamples for all five types of brick.

As would be expected, selecting the C/B ratio of a particular type of brick also selects the water absorption within rather narrow limits. Table III gives the data for C/B ratio, water absorption by 24-hr. cold immersion and water absorption by 5-hr. boiling in terms of average, maximum and minimum for all 12 subsamples of brick C. The data for brick C are selected because they represent the greatest range in C/B ratio of the five types of brick used.

Subsamples 1 and 11 of each sample were tested also for flexural and compressive strength and the results, together with the absorption data, are summarized in Table IV. It will be noted that subsample A-11 is lower in strength than subsample A-1. Bricks B, C, D and E show higher strengths for their subsample 11 compared with subsample 1. That the strength tests on these two sets of subsamples were made by different operators may in part explain the difference in results. The question of how comparable these subsamples are will be considered in the discussion of the results of the freezing-and-thawing tests.

TABLE V.—TABULATION OF FREEZING-AND-THAWING TEST METHODS 1 TO 7.

Method	Brick Specimen	Cycles Between Drying	Method of Drying	Time of Soaking After Drying, hr.	Method of Freezing[c]	Criterion of Failure
1	Half	3	Oven[b]	48	Partial immersion in water	10 per cent loss in dry weight
2......	Whole	3	Oven[b]	48	Partial immersion in water	10 per cent loss in dry weight
3......	Whole	No dry[a]	....	....	Partial immersion in water	Inspection
4......	Whole	3	Oven[b]	48	Air	10 per cent loss in dry weight
5......	Whole	No dry[a]	....	....	Air	Inspection
6......	Whole	5	Room	4	Partial immersion in water	Inspection
7......	Whole	5	Room	4	Air	Inspection

[a] The specimens were totally immersed in water at room temperature after every five cycles.

[b] Drying consisted of 24-hr. exposure in air after thawing and 3 days in a gas-fired oven at 105 to 115 C.

[c] In all seven methods, freezing consisted in exposure to the temperature of the freezing chamber (−9 C.) for 20 hr. and thawing consisted in 4 hr. total immersion in water initially at room temperature.

DESCRIPTION OF METHODS OF FREEZING AND THAWING

The seven laboratory methods used in making freezing-and-thawing tests are described in Table V. The numbering of these test methods corresponds to the numbering of the subsamples tested. Thus, samples[5] A-1, B-1, C-1, D-1, and E-1 were all frozen and thawed by method 1. Since method 1 used half brick, the freezing and thawing was applied to the second set of halves, the first set being used for determinations of compressive strength. Sample 8 was exposed standing on edge and resting on the gravel of a tar-gravel roof on one of the Bureau buildings. Sample 9 was exposed beside sample 8 and stood on edge in pans 1 in. in depth. Figure 1 shows the exposure of samples 8 and 9. Sample 10 was exposed on end, half buried in the soil of the "cemetery" exposure plot described in the paper on the Wick Test (8) (see Fig. 2). Samples 11 and 12 had been kept in reserve. After completion of all tests on samples 1 to 7, freezing and thawing by method 1 was repeated on the half bricks constituting sample 11 for the purpose of checking the comparability of the samples. Method 1 has been used since 1930 on the survey samples (4) and was first described in 1931 (3).

The criterion of failure in methods 1, 2 and 4 was taken as 10 per cent loss in the original dry weight of the brick. A corresponding degree of disintegration as judged by visual inspection was taken as cause for rejection in methods 3, 5, 6, 7, 8, 9, and 10. Since, in most cases, failure took place suddenly and completely, the end points in methods 1, 2, and 4 did not necessarily come on multiples of three cycles. The effect of basing rejection on percentages of loss other than 10 will be considered in the section on discussion of data.

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[5] These subsamples of 20 bricks each will be referred to hereafter as "samples."

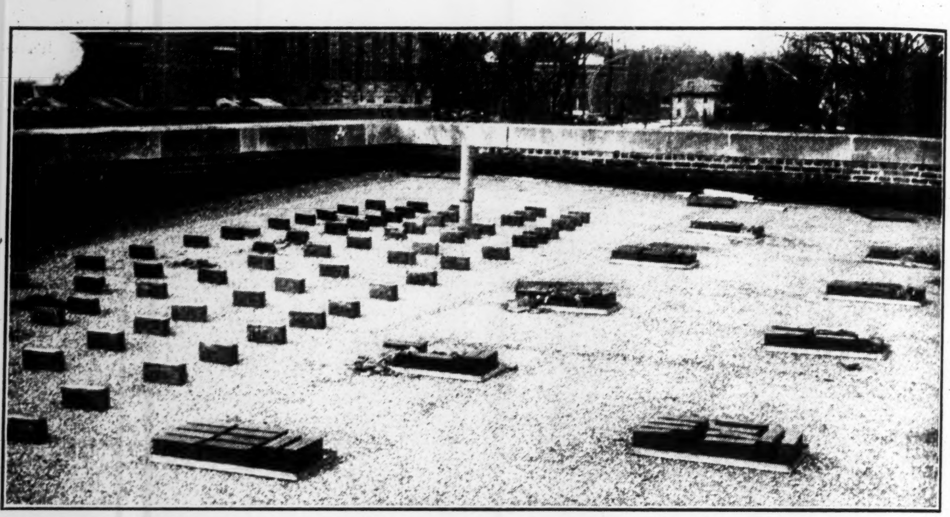

Fig. 1.—View of Exposure Representing Test Methods 8 and 9. The five rows of bricks on the left are exposed according to method 8. The bricks in pans represent method 9. The photograph was taken March 22, 1938.

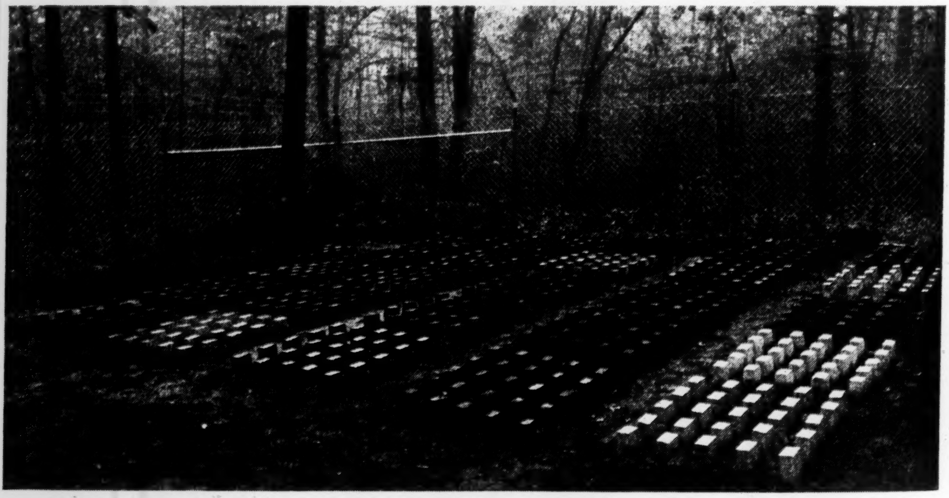

Fig. 2.—View of Exposure Representing Test Method 10. The photograph was taken November 26, 1936.

Freezing and thawing (by methods 1 to 7) was discontinued after 75 cycles.

Certain auxiliary tests will be mentioned and described during the discussion of results.

PRESENTATION OF DATA

Table VI gives the results of freezing and thawing by methods 1 to 7 on the individual bricks of the samples representing bricks A to E. The numbering of the individual specimens is in the order of their failure. This numbering correlated well with the numbering in order of decreasing C/B ratio. In other words, the lower the C/B ratio the greater the resistance to failure in freezing and thawing.

TABLE VI.—RESULTS OF FREEZING-AND-THAWING TESTS BY METHODS 1 TO 7.

The figures opposite the specimen numbers represent the cycle on which failure took place.

Specimen	Method 1	Method 2	Method 3	Method 4	Method 5	Method 6	Method 7	Method 1	Method 2	Method 3	Method 4	Method 5	Method 6	Method 7	Method 1	Method 2	Method 3	Method 4	Method 5	Method 6	Method 7
	BRICK A							BRICK B							BRICK C						
No. 1........	All specimens passed 75 cycles	All specimens passed 75 cycles	19	12	9	19	8	1	1	1	5	3	1	4	2	1	2	11	5	2	5
No. 2........			22	13	9	31	8	1	2	1	7	4	1	5	2	1	2	11	7	2	5
No. 3........			29	13	10	31	8	1	2	1	10	4	1	7	2	2	2	11	10	2	5
No. 4........			34	13	10	32	8	2	2	2	10	4	1	8	2	2	2	12	11	2	5
No. 5........			34	31	10	32	8	2	2	2	10	4	2	8	2	2	3	14	18	2	5
No. 6........			35	35	17	41	8	2	2	2	10	5	2	8	3	2	3	14	18	2	7
No. 7........			38	36	17	43	9	2	2	2	11	5	2	11	8	2	3	18	18	3	7
No. 8........			39	40	17	47	9	2	2	2	16	5	2	13	8	2	3	24	24	3	24
No. 9........			41	46	18	49	9	2	3	2	18	5	2	15	8	6	3	24	24	21	53
No. 10........			46	46	19	53	17	2	4	3	18	7	3	20	31	6	10	31	43	21	....
No. 11........			50	46	24	....	17	2	5	3	21	9	3	22	57	11	17	31	57	56	....
No. 12........			50	46	24	....	18	4	7	3	35	9	6	22	....	58	17	....	73	59	....
No. 13........			52	46	26	....	23	5	7	8	35	15	6	22	....	....	23	....	....	67	....
No. 14........			57	47	26	....	23	9	9	9	54	15	7	29	....	....	29	....	....	....	....
No. 15........			62	47	27	....	25	10	13	12	63	15	7	30	....	....	47	....	....	....	....
No. 16........			62	48	27	....	26	10	13	17	....	15	9	30	....	....	62	....	....	....	....
No. 17........			....	48	29	....	27	10	14	20	....	30	13	30	....	....	65	....	....	....	....
No. 18........			....	48	31	....	27	10	64	22	....	30	13	49	....	....	....	....	....	....	....
No. 19........			....	48	38	....	30	13	....	28	....	44	18	63	....	....	....	....	....	....	....
No. 20........			....	51	38	....	30	53	....	37	....	44	40	....	....	....	....	....	....	....	....
Number of specimens passing 75 cycles.......	20	20	4	0	0	10	0	0	2	0	5	0	0	1	9	8	3	9	8	7	11
	BRICK D							BRICK E													
No. 1........	3	8	6	13	26	8	27	3	3	4	12	11	6	24							
No. 2........	7	8	6	14	26	9	33	4	5	4	18	21	6	24							
No. 3........	8	8	6	24	26	9	36	5	5	4	18	22	8	28							
No. 4........	11	14	7	28	28	10	41	5	5	4	25	24	9	28							
No. 5........	12	15	8	30	28	10	41	5	5	5	25	28	11	29							
No. 6........	15	16	10	40	32	10	45	6	6	5	43	28	11	34							
No. 7........	16	17	11	56	32	12	45	6	7	7	48	30	12	38							
No. 8........	18	18	13	57	32	14	47	6	8	7	55	31	12	39							
No. 9........	19	19	14	57	32	14	47	8	9	9	....	32	14	39							
No. 10........	19	19	14	57	38	14	48	8	10	10	....	34	16	42							
No. 11........	20	24	14	62	38	19	49	12	10	10	....	36	18	44							
No. 12........	20	24	14	....	40	19	52	12	10	10	....	36	24	50							
No 13........	21	26	15	....	40	22	58	12	13	11	....	41	33	51							
No. 14........	23	26	15	....	41	22	58	12	16	12	....	41	42	51							
No. 15........	24	26	17	....	41	23	58	20	19	16	....	41	42	64							
No. 16........	26	30	18	....	41	26	58	27	21	21	....	41	42	....							
No. 17........	30	33	19	....	43	28	75	34	....	21	....	41	50	....							
No. 18........	56	35	22	....	45	28	75	48	....	28	....	43	51	....							
No. 19........	......	39	23	....	45	39	....	....	....	28	....	52	....	....							
No. 20........	......	......	37	....	49	45	....	....	....	46	....	52	....	....							
Number of specimens passing 75 cycles......	2	1	0	9	0	0	2	2	4	0	12	0	2	5							

Figure 3 presents the data graphically for bricks B, C, D, and E in terms of percentage of the composite sample surviving each cycle of freezing and thawing.

Results on the weathered samples

(methods 8, 9, and 10) are given in Table VII.

Discussion of Results of Laboratory Tests

Comparison of the Seven Laboratory Methods:

Since methods 1 to 7 (Table V) can be classified in several ways, some discussion of these methods is warranted as a preliminary to the consideration of the results shown in Table VI and in Fig. 3. Methods 1, 2 and 4 permit no increase in saturation beyond that resulting from three cycles. Methods 6 and 7 partially check the build-up of saturation by air drying at room temperature after every fifth cycle. Methods 3 and 5 permit saturation to build up without hindrance. Methods 1, 2, and 4 start each set of three cycles with 48-hr. saturation by complete immersion in water at room temperature. Four hours complete immersion before starting each set of five cycles characterizes methods 6 and 7.

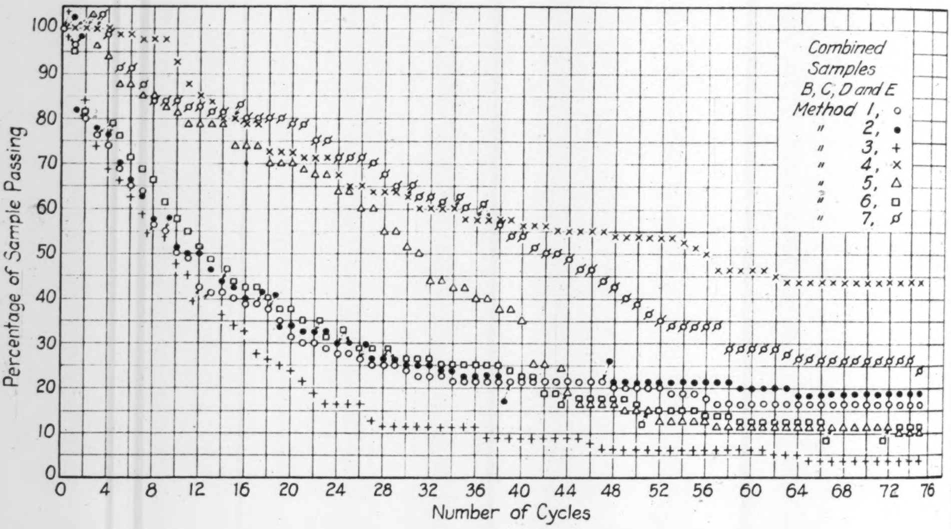

Fig. 3.—Percentage of Each Composite Sample of Bricks B, C, D and E Surviving Given Number of Cycles of Test Methods 1 to 7. Eighty bricks constitute a sample.

The effect of change in a single factor is shown by the following comparisons. Methods 1 and 2 are alike except for the size of the test specimen (half bricks compared with whole bricks). Methods 2 and 4 differ only in that method 2 uses freezing in contact with water and method 4 uses freezing in air. The effect of partial water immersion against air freezing is also given directly by comparing method 3 with 5 and method 6 with 7. Comparing method 2 with 3 and method 4 with 5 shows the effect of checked and unchecked saturation. Method 6 may be regarded as intermediate between 2 and 3 in this respect and method 7 belongs between 4 and 5.

Table VIII was constructed as follows: The data of Table VI were plotted as cumulative percentage of each sample failing on each cycle of freezing and thawing by each method. The area under each curve was calculated and expressed as a percentage of the total area (100 per cent of sample by 75 cycles). This area under the curve of cumulative failures can be considered as measuring the severity of the action of a particular method of freezing and thawing on a particular sample.

Discussion of Effect of Methods on Types of Brick:

(*a*) *Brick A.*—From Table VIII it is evident that the selective action of the seven methods of freezing and thawing on brick A is quite different from the action of these same methods on the other kinds of brick. Freezing in air (methods 4, 5, and 7) is definitely more severe when acting on brick A, than is freezing in contact with water. The reverse of this is true for bricks B, C, D, and E. The reason why brick A behaves as it does is not known. The authors suggest the following as one explanation:

Table VII.—Comparison of Results of Weathering Exposure (Methods 8, 9, and 10) with Results of Laboratory Freezing-and-Thawing Tests (Methods 1 to 7).

Placing of specimens constituting samples 8, 9 and 10 was completed November 1, 1936. This table presents the results of an inspection made on March 12, 1938.

Sample	Method of Exposure	Number of Specimens Passing	Number of Specimens Failed	Number of Cycles of Each Method Which Gave the Same Number of Failures						
				Method 1	Method 2	Method 3	Method 4	Method 5	Method 6	Method 7
A-8	8	20	0	75	75	18	11	8	18	7
A-9	9	20	0	75	75	18	11	8	18	7
A-10	10	20	0	75	75	18	11	8	18	7
B-8	8	12	8	1	1	1	15	4	1	12
B-9	9	6	14	8	8	8	53	14	6	28
B-10	10	1	19	12	[a]	27	[b]	43	17	62
C-8	8	17	3	1	1	1	10	9	1	4
C-9	9	10	10	30	5	9	30	42	20	75
C-10	10	10	10	30	5	9	30	42	30	75
D-8	8	20	0	2	7	5	12	25	7	26
D-9	9	10	10	18	18	13	56	37	13	47
D-10	10	5	15	23	25	16	[c]	40	22	57
E-8	8	20	0	2	2	3	11	10	5	23
E-9	9	12	8	5	7	6	54	30	11	38
E-10	10	2	18	47	[d]	27	[e]	42	50	[e]

[a] At 75 cycles, 2 specimens survived and 18 had failed.
[b] At 75 cycles, 5 specimens survived and 15 had failed.
[c] At 75 cycles, 9 specimens survived and 11 had failed.
[d] At 75 cycles, 4 specimens survived and 16 had failed.
[e] At 75 cycles, 5 specimens survived and 15 had failed.

Dry-press bricks are characterized by relatively large pores in comparison with bricks formed by the soft-mud and stiff-mud processes. Capillary suction, as defined by Westman (9) is less for materials with large pores. It has been observed that freezing of the exposed surfaces of bricks, subjected to freezing temperature while in contact with water, will occur before the water in the pans freezes solid. A possible explanation of the relative severity of air freezing on dry-press bricks is that air freezing seals all six faces of the specimen simultaneously and thus retards the escape of water. Freezing in contact with water apparently leaves the lower face of the specimen temporarily unfrozen and provides a "safety valve" for release of pressure formed by the expansion of water into ice during the early stages of the freezing process.

Table VIII.—Severity of Methods 1 to 7 Acting on Samples of Bricks A to E.

100 per cent represents failure of entire sample on first cycle. 0 per cent represents passing 75 cycles by all specimens of sample.

Brick	Failure, per cent							
	Method 1	Method 2	Method 3	Method 4	Method 5	Method 6	Method 7	Average
A	0	0	36	51	73	25	79	38
B	92	81	90	54	83	92	69	80
C	47	54	66	42	46	50	38	49
D	68	69	82	26	53	76	32	58
E	76	71	84	25	57	64	37	59
B, C, D and E combined	71	69	81	37	60	70	44	62
Avg. A - E	57	55	72	40	62	61	51	57

Brick A provides a striking illustration of the relativity of measures of durability. By methods 1 and 2 all specimens of both samples survived 75 cycles of freezing and thawing. Actually the loss in weight averaged 1.2 per cent with a maximum of 3.5 and a minimum of 0.5 per cent for sample A-1, and for sample A-2 the average loss in weight was 1.3 per cent with a maximum of 4.4 and a minimum of 0.3 per cent. Compare these results with those obtained by the use of method 7. All specimens of sample A-7 failed before the completion of 30 cycles, using as a criterion of failure a loss of 10 per cent of the original dry weight.

Underburned dry-press brick differ characteristically from bricks formed by the soft-mud and stiff-mud process in their manner of failing. Dry-press bricks show little effect from freezing and thawing until very close to the failing point. A progressive weakening of the bond between the clay grains appears to take place. Rather suddenly, then, the brick structure crumbles. The A samples, as would be expected from their narrow range of C/B ratios, are more homogeneous than the other bricks. Method 7 (the most severe) produced no failures on bricks A before the eighth cycle, and all of the sample had failed at the completion of 30 cycles. Considering all methods of tests, brick A is the most durable of the five types tested.

Though not evident in Table VI, methods 1 and 2 did affect brick A. The loss in compressive strength, shown by comparing the strength of the half bricks after 75 cycles with the strength of the corresponding set of halves that received no freezing, averaged 19.8 per cent, with a maximum loss of 30.8 and a minimum loss of 3.6 per cent.

(*b*) *Brick B.*—The samples of brick B have rather low resistance to all seven methods of freezing and thawing. Freezing in contact with water is more destructive than freezing in air. Build-up of saturation adds to the severity of method 7, especially in the later cycles. Methods 1 and 2 are actually in better agreement than the percentages shown in Table VIII would indicate. The correlation is excellent except for the last three specimens (see Table VI).

(*c*) *Brick C.*—Brick C presents a peculiar distribution of properties in its samples. As would be expected from its wide range in C/B ratio (Table II), the C samples are characterized by being very non-resistant at one end of this distribution and highly resistant at the other end. Specimen 12 of sample C-1 (Table VI) lost 30.8 per cent in compressive strength compared with the compressive strength of the corresponding half brick tested before freezing. Specimens 18, 19, and 20 of the same sample showed gains in compressive strength after 75 cycles of freezing and thawing. This combination of low and high resistance to disintegration among the specimens constituting the samples of brick C results in differences in the effects of the different methods being apparent in the middle of the range rather than at either end. The total number of specimens surviving 75 cycles of freezing and thawing appears to be as good a measure of the relative severity of the several test methods as any. Methods 1 and 2 do not differ significantly from each other. Method 3 is notably the most severe and method 7 the least severe. Freezing in contact with water is more effective than freezing in air for all comparisons (methods 2 with 4, 3 with 5 and 6 with 7). Unlimited build-up of saturation adds to the disintegrating effect in the later cycles.

(*d*) *Bricks D and E.*—The samples of bricks D and E did not differ sufficiently in their behavior to warrant separate discussion. Methods 1 and 2 agree. Freezing in water is more severe than freezing in air. Other things being equal, build-up of saturation adds to the severity. The distribution of both the D and E bricks is such that these several relations are nicely illustrated. The samples are neither too nonresistant (brick B) nor too durable (brick A) for a study of test methods.

(*e*) *Composite Sample B to E.*—Values for the samples representing bricks B, C, D and E were combined and the results presented in Fig. 3 on the assumption that a composite sample will tend to smooth out differences caused by variations in the individual samples. Brick A is not included in this composite sample since it reverses the trends shown by the other bricks.

TABLE IX.—EFFECT OF WETTING AND DRYING (ACCORDING TO THE SCHEDULES OF METHODS 3, 5, 6 AND 7 BUT WITHOUT FREEZING) IN INCREASING THE RATIO OF WATER ABSORPTION BY COLD IMMERSION TO WATER ABSORPTION BY 5-HR. BOILING.

The figures in parenthesis represent the cycle number giving the maximum ratio.

Brick	Method 3		Method 5		Method 6		Method 7	
	Initial Ratio	Maximum Ratio	Initial Ratio	Maximum Ratio	Initial Ratio	Maximum Ratio	Initial Ratio	Maximum Ratio
A.....	.....	.....	.....	.....	0.855	0.908 (50)	0.857	0.878 (66)
B.....	.....	.....	.....	.....	0.815	0.844 (4)	0.810	0.827 (47)
C.....	0.760	0.858 (65)	0.640	0.699 (32)	0.630	0.694 (35)	0.635	0.835 (44)
D.....	0.885	0.986 (41)	0.900	0.946 (41)	0.825	0.964 (11)	0.885	0.919 (27)
E.....	0.865	0.979 (41)	0.870	0.930 (23)	0.870	0.932 (11)	0.865	0.876 (28)

Methods 1, 2, 3, and 6 have in common the factor of freezing in contact with water, and in Fig. 3 it will be noted that methods 1 and 2 are in excellent agreement throughout. Method 3 (freezing in contact with water, no drying) duplicates methods 1 and 2 up to cycle 16. Thereafter method 3 gives a smaller percentage of survivals. Method 6 is slightly less severe than methods 1 and 2 up to cycle 16, but, starting with cycle 42, appears slightly more severe. The differentiation between method 6 and methods 1 and 2 may not be significant.

Effect of Degree of Saturation:

The data illustrated in Fig. 3 emphasize the effect of degree of saturation at the time of freezing. That the percentage of absorption of water is a function of time of total immersion is well known (**10**, **11**, **12**). The plan of the present investigations had included weighing representative bricks of each type at various stages of each method of test. Since most of the specimens soon showed disintegration, this was abandoned. Table IX presents some data obtained by the following method: The original absorption by 24-hr. cold immersion and by 5-hr. boiling being known, the bricks were weighed following periods of total immersion corresponding to the "thawing" of methods 1 to 7. In these tests no freezing took place. Instead of being put in the freezing chamber, these specimens were kept at room temperature, either in air or in contact with water, for the same period and as specified for those specimens actually frozen and thawed by methods 3, 5, 6, and 7. These four methods produce a considerable increase in absorption for the first few cycles. Absorption thereafter tends to increase but slowly. Alternations of total and partial immersions (method 3) were most effective in raising the ratio of cold absorption to absorption by 5-hr. boiling. Exposure to air with no partial immersion and with total immersion limited to the 4-hr. "thawing" period (method 7) affected this ratio least. Methods 5 and 6 were intermediate and, on the average, were alike.

In so far as similar tests were made on bricks actually frozen and thawed, the same effect was found. It cannot be assumed that the data of Table IX would quantitatively check similar data obtained by weighing bricks actually frozen and thawed. Temperature and humidity affect evaporation and, with certain types of bricks, an appreciable amount of water is lost by extrusion of ice crystals from the pores during freezing.

Effect of Various Criteria of Failure:

Table VI represents data resulting from using as the criterion of failure a 10-per-cent loss in the dry weight of the specimens or an equivalent disintegration as judged by visual inspection. The effect of using losses in weight other than 10 per cent is shown in Table X.

Taking 3 or 5 per cent instead of 10 per cent loss in weight as the criterion of failure, makes little difference in rating except in the case of brick B tested by method 4. Reference is made to TIBM-57 (13) for a discussion of grading the degree of disintegration by visual inspection.

Comparability of Samples:

Test methods 1 and 2 differ only in their use of half bricks *versus* whole bricks. As previously stated, the results of these two methods are in close agreement, the deviation appearing to be random. The agreement between the sets of samples tested by methods 1 and 2 provides evidence that these sets of samples are comparable. Further evidence is provided by the results of 21 cycles of freezing and thawing applied to sample 11 using method 1. A comparison of the physical properties of samples 1 and 11 is given in Table IV. Twenty-one cycles by method 1 on samples 1 and 11 warrant the following conclusions: The agreement between samples 1 and 11 of bricks A, B, C, and D is as good as or better than the agreement between samples 1 and 2 of the same bricks. Sample E-1 checked the first six specimens of E-11 but thereafter large differences were shown. Except for brick E, the number of specimens of each pair of samples surviving 21 cycles agreed perfectly.

DISCUSSION OF RESULTS OF EXPOSURE TESTS

The bricks tested by method 8 received water only during a rain and the exposure was such that relatively rapid drying followed. Method 9 undoubtedly provided a higher initial saturation of the bricks than did method 8 and the saturation probably did not decrease until the water in the pans had completely evaporated. Method 10, especially during the winter and spring, provided high and continuous saturation of the test specimens. That the degree of saturation at the time of freezing has a marked effect on the durability of bricks is shown clearly for bricks B to E by Table VII. Brick A is, to date, unaffected by any one of these three exposures.

TABLE X.—NUMBER OF SPECIMENS IN EACH SAMPLE PASSING 75 CYCLES OF FREEZING AND THAWING USING 10, 5, 3 AND 1 PER CENT LOSSES IN DRY WEIGHT AS THE CRITERION OF FAILURE FOR METHODS 1, 2 AND 4.

Brick	Method 1				Method 2				Method 4			
	10 per cent Loss	5 per cent Loss	3 per cent Loss	1 per cent Loss	10 per cent Loss	5 per cent Loss	3 per cent Loss	1 per cent Loss	10 per cent Loss	5 per cent Loss	3 per cent Loss	1 per cent Loss
A......	20	20	19	11	20	20	19	8	0	0	0	0
B......	0	0	0	0	2	2	0	0	5	5	1	0
C......	9	9	8	7	8	8	8	6	9	9	9	6
D......	2	2	2	2	1	1	1	0	9	9	9	7
E......	2	2	1	0	4	4	4	4	12	11	10	7

From Weather Bureau records, the

period from November 1, 1936 to March 12, 1938, provided 105 cycles of freezing and thawing, if an air temperature of less than 32 F. is taken as a freeze and an air temperature of greater than 32 F. represents a thaw. The number of freezes occurring within 48 hr. after a rain was 64 for this same period. Since these records represent air temperatures and were measured over a mile away from the "cemetery" and at a different elevation, they may be subject to large error as applied to method 10.

Brick A being unaffected by any of the three types of exposures, no comparisons are afforded between these exposures and the seven laboratory methods of freezing and thawing beyond noting that methods 1 and 2 also failed to disintegrate any of the specimens of brick A during 75 cycles. Method 8 is notably less severe than any of the other methods when used on bricks B to E. If it be assumed that 64 cycles represent the number of effective cycles (freezes and thaws in the presence of water) acting upon sample 9, for the period from November 1, 1936, to March 12, 1938, a direct comparison can be made between method 9 and methods 1 to 7 by noting the number of cycles of the first seven methods which cause the same number of failures of specimens in their samples as was caused by 64 cycles on samples 9. On this basis, method 9 most nearly resembles method 4 in its results on bricks B, D, and E. For brick C, method 9 best corresponds to method 7. Method 7 in general most nearly resembles method 10 in its effect on these same four kinds of brick. Again, if it is considered that 64 cycles represented the effective cycles of freezing and thawing resulting from exposure according to method 9, it is obvious that a considerably smaller number of cycles was effective for exposure 8. On the other hand, method 10 probably subjected its bricks to the full 105 cycles with the bricks relatively well saturated. The five footnotes to Table VII, which call attention to the fact that 75 cycles of certain laboratory methods do not equal the effect of the exposure, apply in all instances to exposure 10.

STANDARDIZATION OF THE FREEZING-AND-THAWING TEST FOR BUILDING BRICK

The effect of intensity and speed of freezing has not been considered. It is possible that differences in the technique of thawing might affect the results. Is the anomalous behavior of brick A characteristic of all dry-press brick? These are some of the things which should be investigated before settling upon any one method as standard. Attention, for the present, will be confined to considering the arguments for and against the several methods here reported.

The arguments for method 1 are: (1) the method is as severe as any (excepting results on brick A) for the first 12 cycles; (2) using as the test of failure the result of weighing the dry specimen instead of visual inspection, eliminates personal judgment; (3) the use of a half brick saves space and permits the use of the other half for comparison and for other tests including strength determination. The objection to method 1 is its slowness. Three cycles in 10 days represent the schedule followed.

Argument 3 does not apply to method 2. The objection to method 1 also applies to method 2.

Method 3 checks methods 1 and 2 for the first 12 cycles and thereafter is more severe. It is speedy, permitting 10 cycles in two weeks compared with 3 cycles for methods 1, 2, and 4 in 10 days. It may be argued that method 3 represents too great a departure from any probable condition of natural exposure in that it represents continuous contact with or immersion in water. However, some retaining walls approach this condition and it is obvious that bricks passing a severe test will be satisfactory under less severe exposures. Method 3 involves the judgment of the operator in deciding when failure takes place.

Method 4 avoids the uncertainty of visual inspection and may be considered as somewhat simulating natural conditions of exposure. When compared with the other methods, however, it is relatively ineffective in producing disintegration (bricks A and C excepted) since it has lowest "efficiency" (see Table VIII) of any of the seven methods used on bricks B, D and E. Method 4 is not recommended because it might pass bricks known to be unsatisfactory.

Method 5 is not recommended if the test is limited to 25 cycles or less. The discussion of method 3 applies to method 5 except for the factor of delayed effect.

Method 6 could be substituted for method 2 without significant change in results. The advantage is its greater number of cycles per month. Its disadvantage is the reliance upon visual inspection.

Method 7 is the most effective on brick A and the least effective on brick C. On bricks B, D and E, method 7 is the least effective of all the seven laboratory methods except method 4. It probably most nearly simulates natural conditions of exposure. Air drying, as exemplified by methods 6 and 7, would seem objectionable for a standard method unless temperature, humidity and wind velocity were controlled during the drying periods.

Method 8 corresponds to the condition obtained by exposing the manufacturer's stock pile in the open.

Method 9 appears to describe the same stock pile stored on an undrained base.

Method 10 is recommended to brick manufacturers for the purpose of checking their own grading of their product. The utilization and interpretation of this test has been ably described by Butterworth (14).

The data of this paper afford some justification for the belief that 50 cycles of an initially severe test are sufficient. Methods 1, 2, 3, and 6 have eliminated at 50 cycles nearly all specimens which did not pass 75 cycles. Methods 1 and 10 have been applied to a number of types of bricks, other specimens of which are now undergoing exposure as panels or walls with a variety of protections. When these results become available, the question of standardizing freezing and thawing can be approached with more confidence.

Summary and Conclusions

This paper reports the effect on the resultant disintegration of certain selected samples of brick, of variations in the method of making freezing-and-thawing tests in the laboratory. Other sets of these samples were exposed to the action of weather, using three types of exposure.

From the data herein reported, the following conclusions are believed justified:

1. The nature of the brick influences the relative effect of differences in the methods of test. Gross error may result from attempts to express results of freezing and thawing by one method in terms of results obtained by another method unless samples of the same bricks have been tested by each method.
2. The use of whole or half bricks as test specimens did not significantly affect the result of the same method of freezing and thawing.
3. With the exception of one type of brick, methods which allowed the saturation of the sample to increase were more severe than methods which reduced saturation by drying after every three cycles. This increased severity

was usually apparent during the later cycles.

4. If oven drying had any disintegrating effect on the brick samples, it was masked by its effect in lowering saturation.

5. With the exception of the same type of brick mentioned in conclusion 3, freezing in contact with water was more severe than freezing in air. This increased severity was most noticeable in the early cycles.

6. The action of both laboratory freezing methods and natural exposures can be summed up by stating that the greater the degree of saturation at the time of freezing, the more effective was the method in producing disintegration.

Acknowledgments:

The authors acknowledge their indebtedness to the several brick manufacturers who supplied samples of salmon bricks at the Bureau's request. Mr. R. H. Brink made the comparative tests on sample 11.

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ADHERENCE OF ORGANIC COATINGS TO METALS

By A. E. Schuh[1]

Synopsis

Organic finishes, in order to be serviceable, must adhere well for protracted periods of time to the underlying base material. It is a well-known fact that finishes vary considerably in this respect. Evidence of poor adherence, such as flaking, peeling, easy removal by some external force, etc., is so glaringly conspicuous, as to suggest a readily assignable cause for the phenomenon. Actually however, these final evidences of adherence loss are in most cases the net result of a prolonged tug-of-war among a large number of competing forces. Although the final sign of adherence loss may be the same for several different finishes, the cause and the road to failure for each may be quite different. In this paper it is hoped to point out and discuss a number of the factors that affect the adherence of different organic coatings. Some of these factors are: specific adhesion, physical state of the surface, chemical state of the surface, composition and structure of the organic coatings, and their degradation with age.

Specific Adhesion

The maximum adhesional bond which can be developed between an organic coating material and the substrate to which it is applied is determined by forces residing at the interface between the two materials. These forces determine the specific adhesion between a given surface and a specific finishing medium. The distance over which such forces can act is necessarily very short, being at most of the order of several molecular layers of constituents in the coating medium. Their sphere of action probably drops to a negligible quantity at distances of the order of a few hundred Ångström units from the substrate surface. Such a distance comprises only about one thousandth of the thickness of an average single-coat paint film. Experimental methods which permit the study of molecular surface structure are necessary for a critical examination of specific adhesion. For this study, the electron diffraction beam is proving to be useful. In the normal coating of supposedly clean, bare metals by paint media, actual metal surfaces are probably never contacted, since most metals are covered with a thin layer of surface compounds, such as oxides, carbonates, etc. The physical and chemical character of this invisible layer sets the limit of realizable adhesion in an organic finish.

Every organic coating material, whether oil-paint, varnish, lacquer, or asphaltic material, consists of a complex mixture of diverse kinds, sizes and shapes of molecules. This heterogeneity is essential to inhibit crystallizing tendencies and to promote film-forming properties. These mixtures contain molecules or molecular groupings which are varyingly asymmetrical and thus have fields of force along their surfaces. Similarly, the surface of the metallic substrate is inhomogeneous and thus the source for bonding forces and attendant tendencies for alignments to satisfy these forces is provided. The substrate surface is fixed and hence, in order to make such alignment possible, a certain degree of mobility for the adhesive constituents in the organic medium must be provided. This is usually accomplished by the use of solvents, heat, or a combination of the two. However, in the case of drying oil films, the film-forming material itself at the time of application may offer sufficient mobility to permit the desired orientation for developing adhesion. In most instances, heat is definitely beneficial in promoting a high order of adhesion. The added thermal mobility offers increased opportunity for the permanently adhesive components in the coating medium to satisfy the force fields in the substrate surface.

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Although specific adhesion is as yet not subject to direct measurement, a number of other factors, which from a practical standpoint are more important in determining the net adherence of an organic coating over a metal, can be investigated successfully. Such other factors are the physical and chemical character of the underlying surface, compositional factors of the finishing material, and effects of structure and aging of the coatings.

PHYSICAL STATE OF THE SUBSTRATE

For convenience, solid substrates might be classified into those having open, sorptive surfaces and those having dense, compact surface structures. As a rule, a high order of adherence is obtained with solids having sorptive surfaces, because here, provided the surface is even moderately wettable by the painting medium, an opportunity for mechanical interlocking of the finish film with the solid substrate is provided. Fibrous materials, such as textiles, many woods, sorptive inorganic solids, and several chemical films on metals, fall into this class. Even here, however, difficulties are at times encountered, owing to local variations in substrate texture, and owing to subsequent chemical and physical changes at the interface. In general, the ultimate adherence is limited by the cohesional strength of either the finish film or the fibrous surface layer of the substrate. Following are some typical values for the tensile strength of various organic coatings, taken from the work of Nelson (**1, 2, 3**)[2] and his coworkers:

Linseed oil.	30 lb. per sq. in.
White lead paint. . . .	240 to 500 lb. per sq. in.
Zinc oxide paint. . . .	470 to 1000 lb. per sq. in.
Varnishes.	400 to 900 lb. per sq. in.
Nitrocellulose lacquers.	800 to 2800 lb. per sq. in.

In the case of solids having a compact surface structure, for example, metals, glasses, plastics, etc., large variations in the adherence of organic coatings are encountered. In many instances, these differences are associated with differences in surface roughness of the solid. In considering any one given metal, treated in a definite manner, the factor of specific adhesion is no longer a variable. With such a fixed chemical surface, the physical surface area may, however, vary over wide limits and the adhesion will obviously be greater in those cases where more molecular surface is offered per unit macroscopic area. Paint technology has long recognized the value of roughening of metal surfaces to promote adherence of finishes, sand blasting,

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[2] The boldface numbers in parentheses refer to the reports and papers appearing in the list of references appended to this paper, see p. 494.

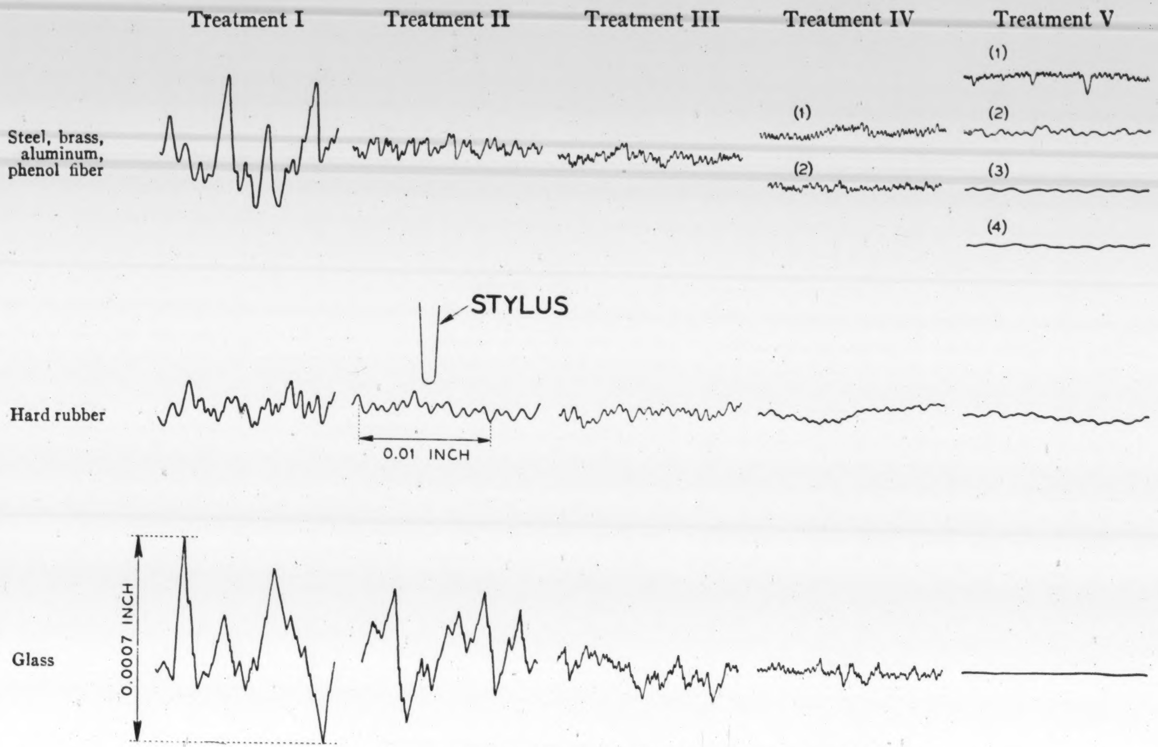

FIG. 1.—Surface Contour of Various Roughened Solids.

Magnification: vertical—4700; horizontal—210. Radius of stylus 0.0005 in.

Treatment I.— 60-mesh steel grit, 30 lb. air pressure
Treatment II.—100-mesh carborundum, 30 lb. air pressure
Treatment III.—180-mesh carborundum, 30 lb. air pressure
Treatment IV.—400-mesh carborundum, 30 lb. air pressure
  (1) Steel and phenol fiber
  (2) Brass and aluminum
Treatment V.—As received
  (1) Cold-rolled steel
  (2) Brass
  (3) Aluminum
  (4) Phenol fiber

shot blasting, or grit blasting, etching, and other methods having been used for many years. Information of a quantitative nature regarding the surface increase by various roughening treatments is therefore of value.

One method of approximating such measurements consists of recording at high magnifications the tracing of a fine-pointed stylus as it rides lightly over the microcontour of a roughened surface. In Fig. 1[3] are shown copies of such tracings over several solids blasted at 15 lb. air pressure with various particle sizes of abrasives. In general, the particle size appears to be the controlling factor in determining the surface contour by a blasting method. As a matter of interest, the contours of roughened hard rubber and glass are also shown and in these two materials, which lie far apart in the range of their elastic limit at room temperature, the tracings are found to be appreciably different. Similar contour tracings have been presented by Darsey (4) for steel surfaces treated in several ways.

In attempting to compute the surface area increase of a roughened metal, as compared with a polished plane surface, it must be recognized that only approximations are possible, since the radius of curvature of the tracing stylus sets the inherent limit of resolution. The point is clearly brought out in the case of polished and polished anodically oxidized aluminum. Contour tracings over these two surfaces were found to be virtually identical, the pores in the anodic oxide coating being too fine to be penetrated by the stylus. According to Rummel (5), the cross-section of such pores is estimated to be 10^{-5} cm. and their spacing at two pores per 10^{-4} cm. With a depth of oxide coating of 10^{-3} cm., the estimated surface increase would be approximately 12.5 times the original surface area. Although the contour tracings showed no surface increase, adhesional strength measurements have shown that organic coatings can penetrate into these pores. There was found a definite increase in joint strength of the same materials when coated on oxidized aluminum. If, however, the oxide coating was sealed in boiling water, the coating constituents could no longer enter the pores and both adherence level and adherence stability were sacrificed to an appreciable extent.

From Fig. 1 it can be seen why the physical roughening of a metal surface is so beneficial for promoting adherence. Surface area increases of at least 25 fold can be estimated in a number of cases. The jagged contour produced by blasting consists of many tiny facets inclined to each other at various angles, which helps to key in the finish by neutralizing stresses normal to the surface. Methods such as blasting serve to clean the metal surfaces very effectively and this may constitute the major factor in improving the adherence of paints to blasted surfaces.

CHEMICAL STATE OF THE SURFACE

If the physical contour of a metal surface is fixed, it is then the chemical nature of the surface which determines the adhesion of a given paint. As previously stated, with a well-defined chemical surface, short range forces set the ultimate limit of adhesion that can be realized. Actually, however, the surfaces to which paints are applied are seldom, if ever, sufficiently well-defined for these molecular forces to act as primary controls of adhesion. In practice, the paint technologist is necessarily and justifiably concerned with grosser aspects of the chemistry of these surfaces. Metals are submitted for application of paints which are covered with macroscopic variant films, usually thousands of molecular layers thick. These layers may be rust, mill scale, other surface compounds, water, dirt, greases, etc. The technical problem therefore is to homogenize the surface as well as is economically feasible by various physical and chemical "cleaning" methods and the importance of such cleaning has been well recognized. Whereas a contaminating layer may itself be well wetted by the paint vehicle and thus be quite adherent to the paint, its own cohesive strength or its adhesion to the base metal is as a rule low, and hence the adhesion of the paint film to the metal is low. Thus by lack of cleanliness a weak link is forged into what otherwise might have been a strong chain.

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[3] The author expresses his thanks to Mr. E. C. Erickson of the Bell Telephone Laboratories for the surface tracings.

Some recent experiments were conducted to ascertain the degree to which a clean metal surface may be contaminated by oils and greases before the adherence of the organic finish is noticeably reduced. The results, still very preliminary, indicate that a contamination of the order of a few tenths of a milligram of grease per square inch of metal surface affects the adherence of most organic finishes. From an engineering point of view this indicates the need for a high order of cleanliness. However, even this small quantity represents several hundreds of molecular layers of contaminant. Undoubtedly the degree of compatibility of the organic contaminant with components of the finishing medium plays an important part. In other cases, selective adsorption of the contaminant may block off the finish medium. It is well known that many of the modern synthetic baking finishes, owing to restricted compatibility with oils and greases, are more sensitive to such surface contaminations than the older type of oleoresinous baking finishes.

Another manner in which the chemical nature of a metal surface influences the adherence of organic finishes is by way of chemical reactions which the base metal can undergo with reactive components in the finish vehicle. This is assuming increasing importance with the wider adoption of new die-cast alloys of such metals as zinc, aluminum, and magnesium. It is quite likely that the initial adherence of organic coatings to clean surfaces of these metals is as high as that obtained with less reactive metals. However, the entry of small quantities of moisture to the metal interface causes difficulties of retaining adherence. The magnesium alloys are particularly difficult in this respect (6) and consequently, chemical pre-treatments are necessary to alter the chemical nature of the surface so that this reactivity is minimized, a dip in a solution containing nitric acid and sodium dichromate having been found effective (7). Other surface films on magnesium have also been investigated, such as fluorides and selenides. Aluminum offers considerably less difficulty in retaining adherence of organic finishes, owing to the compact structure of its readily formed oxide film. However, Edwards (8) has pointed out variations in its alloys, those containing magnesium showing relatively less stability of adherence. For drastic exposures, a heavy oxide film, such as produced by electrolytic oxidation, is recommended and has proven to develop a permanent anchorage. In order to improve the adherence retention of paints over zinc, a number of treatments have been recommended, such as mechanical surface roughening, weathering prior to painting, various chemical conversion treatments, and incorporation in the paint of such pigments as zinc dust to compete with the base metal (9, 10).

During the early drying period of oil

vehicles, a number of products are split off as a result of oxidation, some of which have been identified as short chain organic acids. In the presence of small amounts of moisture which inevitably diffuse to the substrate, reactions with these active metals can occur. These reaction products are less dense than the metal and are weakly coherent. Wing (11) has identified zinc formate produced during the drying of a linseed oil film when coated on zinc. At the Bell Telephone Laboratories it has been found that thick coatings of long-oil varnishes on brass quickly lost adherence when exposed outdoors but retained it indoors. Thin coats of the same varnishes or even thick coats of varnish containing more resin retained adherence even outdoors (12). Baking finishes have shown adherence retention on outdoor exposure when the zinc metal was merely sand blasted, and ordinary paints have given very satisfactory adherence retention when the zinc surface was completely converted to a phosphate. However, complete conversion of the entire surface to these less reactive chemical coatings is important.

Although these more reactive base metals have been selected for discussion, it must not be inferred that iron and steel are immune to these interfacial reactions. The life of painted steel is definitely influenced by the composition of the steel and the chemical state of its surface. The Corrosion Committee of the Iron and Steel Institute (13) is finding that painted copper-bearing steels are corroding more slowly in the atmosphere than other painted steels, as has also been observed by Speller (23). In addition, the importance of complete scale removal is being emphasized in the British studies. It is common practice, where feasible, to convert the iron surface to a phosphate to minimize lateral propagation of rust beneath a paint coating. Special priming paints are widely used. These paints usually contain pigments which prevent or minimize anodic attack of the metal by virtue of their slight alkaline nature or slightly-soluble chromate content. By such measures rust formation or propagation with its attendant destruction of adherence is either prevented or effectively delayed.

COMPOSITION, STRUCTURE, AND AGING OF THE COATINGS

Factors inherently related to the composition of these organic coatings affect their adhesion to metals in several ways. Some affect the initial adherence, whereas others are more related to the retention of adherence.

With respect to initial adherence, most finishing materials, for purpose of application, require solvents as a means of dispersing the permanent constituents. These solvents furnish the required mobility to these constituents of the film to make possible the development of adhesional bonds. In nitrocellulose lacquers particularly, proper solvent combinations are essential for the laying down of continuous adherent films. A number of finishing materials, particularly those containing cellulose derivatives, undergo serious shrinkage as the last part of the solvent escapes. It has been shown (14) that nitrocellulose lacquers contain as much as 10 to 15 per cent of the original solvents at the dry-to-touch stage. In these lacquers and enamels, plasticizers and resins find use not only as a means of providing additional anchorable constituents, but also to provide internal cushioning to counteract shrinkage effects as solvent escapes. A resin-free cellulose acetate film, even though plasticized, when deposited from solution shrinks to such an extent that it will pull itself off spontaneously from almost any dense surface. However, when hot pressed from a dry sheet of acetate of the same composition, the film does not shrink nearly so much and in thin layers can adhere over various surfaces where the same thickness laid down from solution would peel. Similarly, a solution of ethyl cellulose deposits a film of poor adherence on a smooth metal surface; yet, when hot pressed in a dry state a high adhesional bond is developed between smooth metal surfaces.

Permeability

Certain finishing materials, such as drying oils and varnishes, vary considerably in their acidity and hence in combination with certain metals and moisture promote the development of interfacial reaction products which destroy adhesion. The permeability of moisture through organic finish films varies over a wide range depending on the composition and formulation of the finishing medium. A low moisture permeability is important even though the complete exclusion of moisture from the metal-finish interface is beyond the range of practical possibility. This is so because a film of low permeability will retard the entrance of moisture sufficiently to prevent establishment of equilibrium with the external atmosphere during limited periods of high humidity. Reactions at metal surfaces in general exhibit a sharp acceleration in rate at relative humidities of 70 per cent or higher. These accelerations are largely avoided by the use of low permeability coatings.

The following table lists the initial permeability to moisture of a number of finishes. The coatings were deposited at a thickness of 0.001 in. on cellophane and their permeability was determined one month after application, by measuring the transfusion of water vapor at a differential humidity of 90 per cent and constant temperature (25 C.).

Permeability of Moisture Through Organic Coatings

	Milligrams of Water per 0.001-in. Film per Square Inch in 24 hr.
Cellophane (not moisture proof)	300
Cellulose acetate	300
Vinyl acetate	115
Vinyl chlor acetate	27
Linseed oil	80
Long oil varnishes	36 to 48
Long oil varnish plus aluminum powder	14
Short oil varnishes	17 to 24
Single-pigment paints	29 to 56
Nitrocellulose lacquers	15 to 87
Orange shellac	16
Asphaltic coating	5

The wide range of initial values shown in the table is of significance primarily in those cases where a high order of reactivity with the base metal prevails. In more normal cases, the change in permeability with age becomes increasingly important. Most finishing materials gain in moisture impedance on aging up to the point where intrinsic film failures, such as checking, cracking, etc., begin to appear, at which point a ready entrance to moisture may be provided, depending on the number and depth of the cracks developed. The speed at which such intrinsic film defects appear is governed by the composition of the film-forming material, that is, vehicle plus pigment, and the nature of the exposure environment.

Structure and Stability

The composition of the coating material affects adhesion in another manner. Different finishing materials develop coatings of widely differing structure and attendant physical behavior. Some are tough, that is, both strong and distensible, others non-distensible but high in tensile strength, and still others highly distensible but weak. These structural characteristics affect the adherence of the coatings in several ways. In some instances, shrinkage stresses within the coatings may grow to the point where they can overcome adhesion. In others, the apparent loss of adherence may really be attributable to a coherence failure close to the substrate, owing to the low tensile strength or else the brittleness of the coating. Even though it may be questionable to classify this as an adherence failure, from a broader practical point of view such a classification appears justifiable, since with a material of this type the metal base may be readily laid bare to the eye by even the mildest external force. Whether there still remains an exceedingly thin film of the coating over the metal has little practical significance. On aging, many organic coatings fail in just this manner.

For purpose of gaining more insight into this question of structure, a number of different types of finishing materials have been subjected to stress-strain analyses of their free and adherent films. In free film studies, admirably conducted by Nelson and his coworkers (**1**, **2**, **3**), a detached film specimen is subjected to tension under controlled conditions. The load required to stretch the film and the amount of stretch are recorded up to the point of rupture of the film. The stress-strain curves thus obtained are highly informative with respect to the strength of the film, effect of pigmentation, type of flow, effect of environment, etc. By relating such curves to composition of the coating materials and repeating the tests at periodic intervals, a comprehensive understanding of film structure and aging effects is obtained. However, no information with respect to adhesion and its effect on the film characteristics is furnished by the free film method. This has been overcome to a large extent at the Bell Telephone Laboratories by performing various physical tests on the actual finished specimens, that is, with films adherent to the metals (**15**, **16**, **17**). Distensibility is determined either by direct extension of the finished specimen until film cracking occurs, or by bending around suitable mandrels and calculating the film stretch at the mandrel radius at which first cracking occurred (**18**, **19**). The relative film strength is determined by an abrasion test developed at the Bell Telephone Laboratories (**20**). In this test, an abrasive-air stream (180 mesh carborundum—10-cm. mercury air pressure) is allowed to impinge against the surface of the finish, suitably positioned, until the base metal is revealed. The required amount of abrasive is determined and with known film thickness, an abrasion value, in terms of grams of abrasive per 0.001 in. of coating, is obtained. In this test a relative measure of the cohesive strength of the coating is obtained. The abrasion value in effect integrates the area under a stress-strain curve of the detached film. Hence, on dividing the abrasion value by the experimentally determined distensibility of the anchored film, an approximation of the relative, effective tensile strength of the coating is obtained. Just as it is found in free film stress-strain analyses that aging is accompanied by reduction in elongation and increase, frequently followed by decrease, of tensile strength of many finish films, so in testing anchored specimens are similar changes with age revealed. In addition, changes in adherence are measured by noting the velocity of impact of a rotating hammer (**21**) as it strikes the paint coating and removes it from its base metal.

By means of such physical analyses, the various types of organic finishes can be investigated and relations with their adherence properties shown. Thus, linseed oil paint films show in their early age very high distensibility and low tensile strength. The flow is elastic over a wide range of elongation, coatings stretched over 60 per cent of this length having been found after rupture to return to their original length. On aging, that is, on progressive densification of the gel structure, the film gains strength and loses distensibility. As the climatic factors such as sunlight, rain and temperature changes further age the film, it degrades and disintegrates from the outer surface inward, gradually losing both tensile strength and distensibility. Both these aging processes are accompanied by effects on adherence. Initially, with clean surfaces, the adherence is excellent and much greater than the cohesional strength of the film. During early aging, the anchored film develops increasing shrinkage stresses. The magnitude of these stresses is largely determined by the thickness of the coatings. The flaking and peeling of large pieces of paint from structures that have been repeatedly coated is generally familiar. In the later stages of aging, film degradation proceeds to the point where checking and cracking develop and frequently only a mild external disturbance (impact, thermal stresses, etc.) may be required to remove most or all of the coating from the base material. As might be expected, pigmentation of the oil film plays a most important part in these aging phenomena. However, it is beyond the scope of this paper to enter a discussion of pigmentation.

The shrinkage stresses that develop in many organic coatings do not really affect the specific adhesion of the coatings to the base metal, but set up forces opposing adhesion to the point where they become larger than the adhesional forces. A thin coating of gelatin on clean glass adheres well over a wide range of humidity. A thick coating, on the other hand, on drying soon sets up stresses sufficient to pull itself off the glass. The interesting effect is that often at the moment of severing from the glass, the adhesion to the glass is high enough to pull glass away with it, showing that the adhesion was higher than the cohesion of the glass. The phenomenon has actually been commercially exploited to produce a decorative frosted effect on glass.

Certain lacquer coatings have exhibited a similar behavior (**17**). Metal foils covered with them coil into tight spirals as the lacquer coating dries and ages. A thick film of a certain lacquer was found on early aging to bow appreciably a copper panel $\frac{1}{32}$ in. thick. The adhesion must have been very high to bring this about. On outdoor exposure, this material (**12**), when applied more than 0.002 in. thick, spontaneously split off large pieces of coating after only three months. However, the remaining adherent sections of the coating on the test panels withstood the impact of a revolving hammer at 2000 r.p.m. without loosening. A thinner coating (0.001 in.) of the same material is still adherent and in excellent condition after four years on the roof.

A further illustration of these opposing shrinkage stresses is shown in a recent experience with an adhesive joint between two polished steel surfaces. The cementing material was a thermoplastic molding compound. When used as a molding compound, the plastic on cooling parted spontaneously from the mold surface with an unmarred lustrous surface, as would be expected from a satisfactory molding compound. The same dry material, however, when squeezed between two polished steel surfaces into a thin layer, under conditions of heat and pressure comparable to those used in molding, gave a joint strength in tension of about 1000 lb. per sq. in. The explanation for this phenomenon lies in the fact that in a thin layer the shrinkage stresses on cooling were far from sufficient to overcome the adhesion to the smooth metal surface. The same material, when squeezed into a thin layer against finely-roughened steel surfaces, gave a joint strength in tension of 6000 lb. per sq. in., approximating the cohesive strength of the plastic itself.

Shellac is another organic coating material which in thermoplastic joining of metals has given joint strengths in tension of about 2 tons per sq. in. (**22**). However, shellac as a coating material for metals gives a poor account of itself. Its impact and wear resistance are very low. A stress-strain analysis of the material provides the answer. The material exhibits very little distensibility and hence is quite brittle. Even a mild external force is sufficient to shatter the coating and expose the base metal near the point of impact.

Varnishes, depending on the amount of resin incorporated in the heat-bodied drying oil, may act either similar to a drying-oil film or at the other end of the scale like a shellac film. As the amount of resin is increased the material progressively loses distensibility. The abrasion value of a short-oil ester gum varnish (a typical furniture varnish) is usually less than 10 g. per 0.001 in. of coating. Its distensibility is less than 3 per cent. A long-oil varnish, on the other hand, usually shows abrasion values of greater than 50 g. and an elongation of greater than 60 per cent. On exposure, the short-oil material shows cracking and peeling witnin a few months. Even a mild scuffing then removes it from the base. The long-oil material, on the other hand, usually shows excellent adhesion for a long time. Varnishes compounded with modern synthetic resins are not so sensitive to embrittlement as those made with the older types of varnish resins.

Baked finishes in general form strong tough films with a high level of adherence to the base metal. The process of baking definitely increases the strength of bond to the substrate. The baked coatings, being both tough and adherent, are removed mechanically only with difficulty. Round-headed nails finished with certain baking enamels have been driven into pieces of hard oak wood without disrupting the finish (**21**). With increasing coating thickness, shrinkage stresses during baking and subsequent aging are developed at times, which may grow sufficiently to pull off the coating from its base.

CONCLUSION

It is hoped that this survey of several types of organic coatings on metals will have served to indicate the complexity of interrelated factors that influence the adherence of such coatings. It is only in rare cases, usually those of gross negligence, as in improper cleaning, use of excess finish, etc., that a single simple cause may explain a given case of poor adherence. In most other cases, faulty adherence is attributable to complicated causes which frequently are not related to the initial strength of adhesional bond. The coating material may have degraded to such a point of weakness and brittleness that a slight impact or other external stress causes it to fall off. In other instances, a new and weakly-coherent interface may have developed as a result of high moisture permeability and thus destroyed the adherence of the finish. Finally, with thick coatings, sufficient internal stresses may have developed in certain coatings that even the best adhesional bond is overcome and as a result poor adherence of the finish produced.

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DISCUSSION

MR. F. N. SPELLER[1] (*presented in written form*).—The problem of how a metal should be prepared for painting is now generally considered more important relatively than the kind of paint applied, and, therefore, is now receiving more attention. The results of Mr. Schuh's work have gone a long way towards placing the adherence of paints on a practical scientific basis. This paper should be very useful to those who are investigating various phases of this problem such as methods for cleaning and preparing metal surfaces for painting.

However, a perfectly clean metal surface is rarely encountered in practice, and is not necessarily the best for painting. As the author points out, the surface formed by metal films is the one that usually has to be taken into consideration, so that the physical and chemical properties of these films often determine the adherence of paints. Fortunately, much is already known about these surface films mainly from work done on corrosion, and in many cases they can be formed so well as materially to improve paint adherence.

Metal surface films formed in hot solutions constituted the base for the remarkably durable automobile coatings now enjoyed. This principle is also illustrated by some tests of synthetic paints applied to hot-rolled sheets and immersed in natural fresh and sea water for about one year. Most of these paints developed more or less blistering within a few months. The blisters were considerably reduced in size and, in some cases, they were prevented entirely by pretreating the metal for a few minutes at 70 F. with a phosphoric acid chromated solution, and drying without washing at room temperature.[2] When steel is treated in practice with such inhibitors at normal temperature, extra precautions must be taken to insure completion of the reaction, removal of all material that is readily soluble, and complete drying of the treated metal; otherwise eventual failure of the paint may be expected. The practical application on a large scale of inhibitors for this purpose at normal temperatures is still in the development stage.

The author's experimental figures indicate that an asphaltic paint showed the least initial permeability to water of all materials tested. In view of the large amount of coal-tar and asphaltic paint used as primers for hot bituminous coatings, it would be interesting to know how this asphaltic film was prepared and what kind of asphalt was used. Over longer periods, coal tar absorbs less water than asphalt in thicker coatings (accompanying Fig. 1). But single coats of either of these thin primers usually give protection for less than three months in corrosive air and leave much to be desired in this respect.

This paper is worthy of thoughtful reading by everyone interested in organic coatings.

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[1] Advisory Engineer, National Tube Co., Pittsburgh, Pa.

[2] A fuller discussion will be found in a paper by F. N. Speller, "Pre-Treatment of Metal Surfaces for Painting," *Industrial and Engineering Chemistry* (1938).

MR. W. J. CLARKE.[3]—The particular materials referred to by Mr. Speller were a gilsonite varnish and a blown asphalt cut. The method of determining the permeability was the common vapor cup method, using a cellophane base, on which the coatings were laid, and then following the Gardner technique. This method, as run, was not sensitive enough to differentiate coal-tar materials from gilsonite and other very impermeable substances.

MR. C. C. HIPKINS.[4]—Do you recall, Mr. Clarke, about what thickness those bituminous films were?

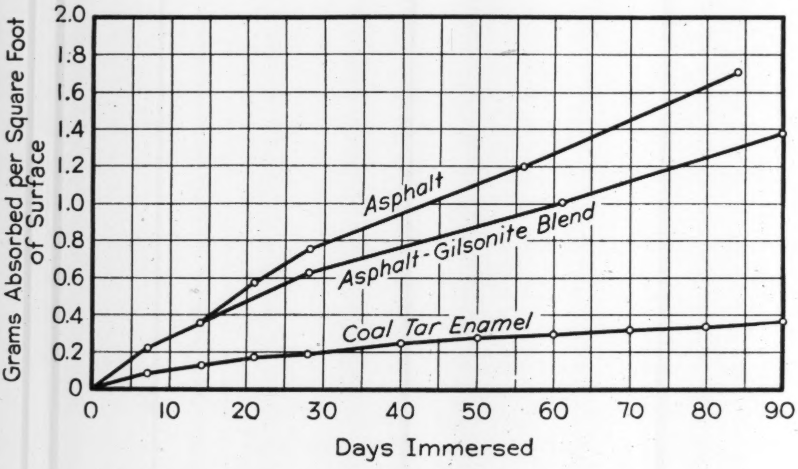

FIG. 1.—Water Absorption of Bituminous Pipe Coatings.

Test made by Guy Corfield, Southern California Gas Co.

MR. CLARKE.—All our thicknesses were 1 mil—that is, all the determinations were for 1-mil coatings on cellophane.

MR. HIPKINS.—That is a very important point. Of course, allowing for the differences between the types of bituminous materials that Mr. Speller could use on pipe, the question of how thick he applied them is very important, as to whether they seal out moisture well or not.

MR. H. A. NELSON.[5]—Mr. Schuh's paper will prove very useful, particularly in focusing attention upon the question of adhesion. Everybody is interested in the subject, and all information and suggestions that can be collected are worth while.

Mr. Schuh has placed some emphasis on preferential absorption of ingredients of the paint film at the metal surface. Most paints are essentially mechanical mixtures that offer plenty of opportunities for preferential absorption, which means that one might find almost anything concentrated at the surface of the metal to modify the interface. The importance of this is undoubtedly illustrated by the very profound adherence changes that can take place when a paint film is baked. In such cases, I suspect that the most important changes are those that involve the particular ingredient that pre-empted the surface while the paint film was still in a more liquid state. In any case, something has to be there and whatever material finally wins out in the contest—volatile thinner, oil, or resin—must have some determining influence on the adherence results. With the rising tide of new developments in resins and oils and their modifications appearing on the scene, some practical means for determining what ingredient is most likely to be concentrated at the interface would be very helpful.

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MR. JUNIUS D. EDWARDS.[6]—In connection with Mr. Schuh's pertinent remarks about the effect of increasing adherence of organic coatings by increasing the area of contact by surface roughening, there is some information in an unrelated art which is of interest. There is a device known as an electrolytic condenser, which comprises an aluminum anode in contact with an electrolyte. The aluminum anode bears an oxide film produced electrolytically and the capacity of the condenser is a function of the thickness and area of this oxide film which serves as dielectric.

Manufacturers of these condensers, in order to get maximum electrical capacity in minimum space, have adopted the expedient of etching the aluminum anode in order to increase the surface area and hence the capacity. By the use of suitable etching procedures it is possible to get as much as 500 or 600 per cent or more increase in the surface area. This fact is of interest at the moment because it suggests a precise method of measuring the increase of surface area on a piece of roughened aluminum. If the oxide coating is maintained at the same thickness in the case of two surfaces, the areas are in direct proportion to the electrical capacities, which can be easily measured.

All of the etched surface may not be available for increasing surface adhesion. The increased area is due to penetrating pits in the aluminum surface and some of the pores may be too small for the coating material to enter. The surface contour may also have an effect on the degree of adherence; under-cut pits are particularly effective in holding a coating. Finally, the surface must be suitably passivated if it is to be most effective in holding organic coatings—particularly under conditions of moist exposure. Surface roughening is most effective in increasing adhesion against the loosening action of vibration and mechanical shock.

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ROSIN CRYSTALLIZATION

A DISCUSSION OF THE THEORY UNDERLYING CRYSTALLIZATION APPLIED TO ROSIN

By E. A. Georgi[1]

Problems involving the crystallization of rosin frequently arise. Comments heard regarding this crystallization indicate there are many who believe that the crystallization of rosin is a mysterious phenomenon about which very little is known. On the contrary, by considering facts already known in the light of physicochemical laws, the crystallization of rosin takes on a fairly rational outlook. At most, the worst which can be said for crystallized rosin is that greater heat and higher temperatures are required to melt it.

Crystallization does not in any way change the chemical properties of rosin. Of course, it is annoying for any user to find that the rosin he has just put in solution is crystallizing out. However, if one has exceeded what we know to be the limit of solubility of the rosin acid, the crystallization is nothing more mysterious than the laws of nature and chemistry forcing themselves to our attention. The following discussion is therefore aimed at outlining some of the laws and facts which govern the crystallization of rosin.

The matter of prevention of crystallization must be the result of research. It should be pointed out, however, that in considering the prevention of crystallization it is probable that more can be done from the standpoint of processing of the rosin than will be accomplished by trying to find a crystallization preventive. A thorough study of the rosin acids, particularly the conditions affecting isomerization, should lead to information telling us how to *produce* a noncrystallizing rosin.

Yet another thing is necessary for the solution of the problem and this is an accurate test program and method for determining the extent to which a rosin will crystallize as well as its tendency to do so.

Rosin is often considered as being a solid because of its apparent rigidity. This, however, is a false concept, since rosin in its usual state is merely a supercooled liquid. Thus it has been shown by numerous publications[2,3] that the physical properties of rosin change continuously with temperature whereas a transition from the solid to the liquid state is always accompanied by a discontinuous change of properties. The solid state is usually evidenced by crystals. These crystals, or crystallization as the process of formation is called, result from the orientation of the molecules, in this instance rosin acid molecules, so that the configuration forms a stable space lattice.

In considering crystallization from a melt (liquid rosin) we can assume that the rosin acid molecules wander about with a random motion. Accordingly there will be a time when several molecules collide in just the right configuration to form a unit crystal or nucleus. In the case of simple materials like metals this chance arrangement is probably quite often attained, but the reverse, the flying apart of these arrangements, is likewise often attained. With a complicated molecule like abietic or pimaric acid the chance of forming nuclei is therefore greatly reduced, but likewise once formed they will be quite stable because of the great number of atoms in the molecule.[4] Now, once having formed a crystal nucleus, if the temperature is below the melting or saturation point, thermal energy will be given off while the crystal grows. This thermal energy is the heat of crystallization and is equal to the heat of fusion which for abietic acid appears to be low—19.4 cal. per g.[5] Actually, in order that the crystals may grow, this heat must be conducted away from the vicinity of the crystal. Thus the growth will be governed by the thermal conductivity and also the viscosity of the melt or solution. We know the viscosity of rosins is high[3] and the thermal conductivity is extremely low (0.0002 g. cal. per sec. per sq. cm. per cm.)[6] so we can expect the rate of crystal growth of the rosin acids to be low. This it is, in comparison with other organic materials such as benzoic acid, etc.

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[1] Experiment Station, Hercules Powder Co., Wilmington, Del.

[2] J. M. Peterson, "Physical Properties of Wood Rosin," *Industrial and Engineering Chemistry,* Vol. 24, p. 168 (1932).

[3] J. M. Peterson and E. Pragoff, "Viscosity-Temperature Relationships of Rosins," *Industrial and Engineering Chemistry,* Vol. 24, p. 173 (1932).

Crystallization is a phenomenon which takes place in two classes of materials: (*a*) supercooled liquids, that is, liquids below the melting point of the solid state; (*b*) supersaturated solutions (we will not consider sublimation). Now since rosin will at times crystallize, we must consider it as being in one of the above two classes. However, regardless of which class we place it in, the results would be the same. We know, for example, that most of the better known (and better characterized) rosin acids melt above 150 C.; therefore, if we consider rosin as a supercooled impure compound, we at once see that the tendency for rosin to crystallize must be present at all temperatures below 150 C. Actually, rosin consists of from 85 to 95 per cent rosin acids, the remainder being a heterogeneous mixture of esters, alcohols and hydrocarbons. Aside from this, we know that more than one rosin acid is present but very little is known about mutual solubility relations. If we consider rosin as a solution of one rosin acid in another and of the mixture of rosin acids in resenes, then the tendency for the rosin to crystallize would be present only when we exceed the solubility of the crystallizing component.

Since crystallization in a given solvent depends on the concentration of solute, we would expect the acid number of the rosin to be related to the tendency to crystallize. That is to say, the more acid we have present in the solution the higher will be the acid number and the greater the tendency to crystallize. Of course, it must be recognized that one rosin acid may act as a solvent for another and hence spoil the relation to acid number. Experience, however, has shown that the solvent power of the resenes for rosin acids is many times greater than the solvent power of one acid for another. This is probably true because of the similarity of structure of the different rosin acids. Of course, the exact history of the rosin must also be considered so that the above remarks apply more strictly only to rosins having a similar history. Referring to Fig. 1 we see three regions marked off by vertical lines. The first

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[4] Davy, "Study of Crystal Structure and Its Applications," McGraw-Hill Book Co., Inc., New York City (1934).

[5] Unpublished data.

[6] H. E. Nash and E. A. Georgi, "Relative Cooling Rates of Crystallizing and Noncrystallizing Rosin," *Transactions*, Am. Inst. Chemical Engrs., Vol. 23, p. 102 (1929).

(solution) means that not enough rosin acid is present to form a saturated solution. Rosins having an acid number below 125 are probably in this region. The fact that a rosin of acid number 125 is a complete solution can be seen from Fig. 2 in which the saturated solution line for rosin of acid number 125 extrapolates to 100 per cent.[7]

The next region, the metastable region, is the region of supersaturated solutions. In this region substances (rosins) can crystallize, but probably will not[8] unless some impetus is received, such as, moisture, dirt, agitation, seed crystals, etc. The acid numbers of rosins in this region are between 125 and 160. In the third region, rosins having an acid number between 160 and 185.6, we may expect spontaneous crystallization at all times under normal conditions, that is during filling the barrel, storage, melting, etc. This, in fact, occurs in such high-grade products as commercial abietic acid. The above remarks apply to rosins having a slightly negative rotation. If the rosin has a dextro rotation, the acid numbers limiting the described regions will be raised. It must be mentioned again, however, that while the above remarks indicate a lessened tendency to crystallize for more dextro rotatory rosins, this is true only for rosins having a similar history and similar properties, such as viscosity, etc. Figure 1, while here presented only as a schematic representation of the tendency for a given rosin to crystallize, has as its basis the crystallization history of over a hundred rosins. Occasionally a rosin is found which does not fit the scheme but invariably such rosins have been given some type of drastic treatment.

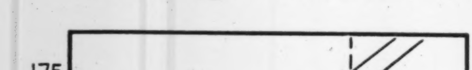

FIG. 1.—Schematic Representation of Relation of Acid Number to Crystallization Tendency.

To return to a consideration of rosin as a supercooled impure compound, we may assume rosin to be a "glass." Glasses have been shown to have certain properties with regard to crystallization. For example, they show increasing speeds of crystal growth or linear crystallization velocity (L.C.V.) as the degree of supercool increases. At some ten or twenty degrees below the melting point the L.C.V. reaches a maximum and thereafter decreases. This drop in L.C.V. is brought about mainly by the rapid increase in viscosity.

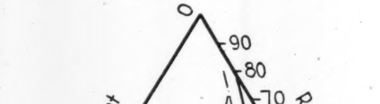

FIG. 2.—Solubility of Rosin in Water-Alcohol Mixtures.

Of most importance for crystallization to take place there must first be formed crystal nuclei. The rate of formation of these nuclei also depends on temperature and, like the L.C.V., passes through a maximum at some point below the melting point of the solid.

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[7] E. A. Georgi, "The Rosin Acids," *Journal of Chemical Education*, Vol. 10, p. 415 (1933).

[8] Tutton, "Crystalline Form and Chemical Constitution," MacMillan and Co., London (1926).

In Fig. 3 are seen curves[9] showing the relation of temperature (of supercooling) and linear crystallization velocity and rate of crystal nuclei formation. The two important points are that the temperature of greatest nuclei formation or greatest *tendency* to crystallize, as well as the temperature of *most rapid crystal growth*, both lie well below the melting point and that the temperature of maximum nuclei formation is lower than the temperature of maximum crystallization velocity.

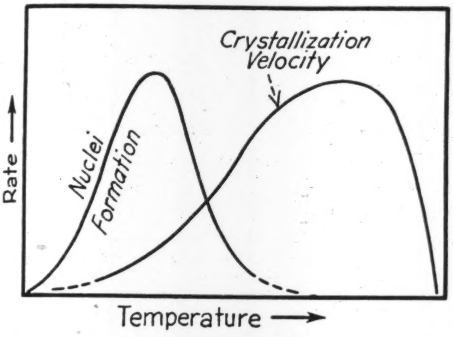

FIG. 3.—Relation of Temperature to Crystallization Velocity and Nuclei Formation.

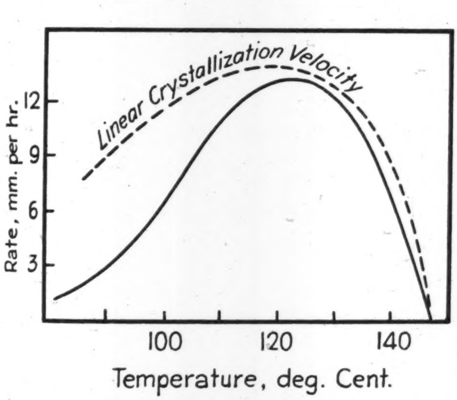

FIG. 4.—Crystallization Velocity *Versus* Temperature of Rosin.

That such a maximum rate of crystallization does exist for rosin (commercial abietic acid) has been proven experimentally and the results are shown in Fig. 4. This curve (solid line) (see Table I) shows that the maximum rate of crystallization is attained at approximately 120 C., that no crystals will form above 150 C., and that the *rate* of *crystal growth* is practically nil below 60 C. The dotted curve (Fig. 4) is a plot of calculated values for the L.C.V. of rosin.[10] The temperatures mentioned will be proportionately lower for less "pure" (lower acid number) rosins.

TABLE I.—DATA OBTAINED BY MEASURING THE RATE OF GROWTH OF CRYSTALS (ABIETIC ACID) IN ROSIN.

Samples contained in tubes and held in constant temperature oven for various lengths of time.

TEMPERATURE, DEG. CENT.	RATE OF CRYSTALLIZATION, MM. PER HR.
80	0.8
90	3.1
95	5.0
100	7.6
105	9.1
110	11.2
120	13.2
125	11.6
130	10.3
135	9.2
145	3.0
149	0.0 (seed crystals dissolve)

Data have not been obtained which would show the *rate* of *crystal nuclei formation* or temperature of maximum nuclei formation for commercial abietic acid, but we can say definitely that it is somewhere below 120 C. The appearance of crystallization nuclei which have grown enormously can be seen in Fig. 5. This shows about twenty-five nuclei in a block of commercial abietic acid. Practically speaking, the above facts mean that there is less tendency for rosin to crystallize on being cooled than on heating a previously cooled sample because of the relative temperatures of the maxima of the linear crystallization velocity curve and the nuclei formation curve, provided, of course, the heating is not done too rapidly or carried to too high a temperature.

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[9] Tammann, "States of Aggregation," D. van Nostrand Co., New York City (1925).

[10] W. Reinders, "Die Krystallizationsgeschwindigkeit aus der unterkühlten Schmelze," *Recueil des travaux Chimiques*, Vol. 51, p. 589 (1932).

Of course, abietic acid is not the only rosin acid present in rosin. In fact, probably very few rosins contain even as much as 50 per cent of abietic acid. We know that while the optical rotation of abietic acid is negative, many isomers could exist which would have a positive rotation. We know also that at least one of these dextro rotatory acids is more soluble in various solvents than is abietic acid.[7] Hence, it is fair to conclude that if rosin is a solution it would take more of this dextro rotatory acid to form a saturated solution than it would abietic acid; consequently, the idea that a high dextro rotation of rosin is desirable because the rosin will have a less tendency to crystallize is at least partially founded on fact. Residue rosins obtained by fractionally crystallizing out abietic acid have been observed to have much higher dextro rotation and lower crystallization tendencies. The fallacy of using rotation as a criterion for crystallization tendency lies in the fact that the rotation which we measure is the net rotation of not only two rosin acids, but others as well as optically-active resenes.

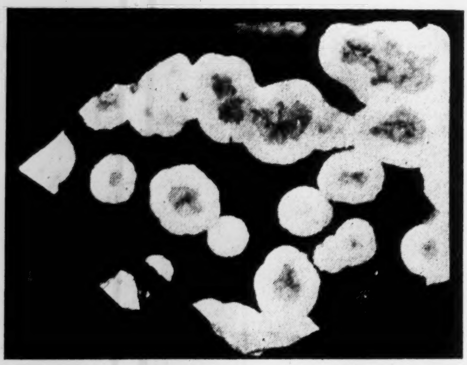

FIG. 5.—Crystal Nuclei in Block of Rosin (One-quarter Natural Size).

While speaking of optical rotation, it is important to point out that for various rotations to be comparable they must have been obtained in an exactly analogous manner. Obtaining rotations directly on the supercooled rosin is perhaps the best way to avoid misunderstanding, since it has been shown, for example, that abietic acid in 10 per cent butyl alcohol solution will have a specific rotation of -60, while in benzene solution it will have a rotation of $+10$.[7] (See Fig. 6.) Ethyl alcohol gives a still greater negative rotation for abietic acid.

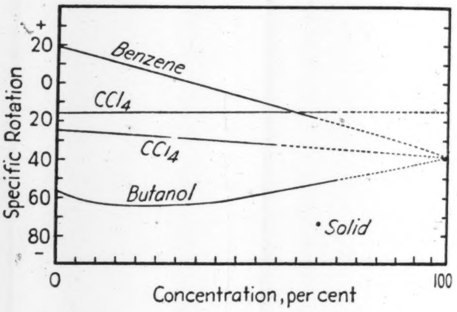

FIG. 6.—Specific Rotation-Concentration Curve of Rosin Acids.

Regarding the tendency to crystallize at any given temperature, it was noted by Tammann that the best way to produce crystallization was to hold the material at a definite temperature gradient. If we consider this fact in connection with Fig. 3, we obtain the results indicated graphically in Fig. 7, which show what will tend to happen if we hold a tube of rosin, one end of which is at the temperature at which crystals cannot exist, the other end of which may be at room temperature. Under these conditions a zone—marked 1–2 in Fig. 7—should tend to develop in the tube. It is to be expected that the width of this zone would be an indication of the tendency of the rosin to crystallize. Such gradients exist both in filling barrels and in melting rosin of large lump size. These gradients for both crystallizing and noncrystallizing rosin have been measured.[6] This work has shown that the heat of crystallization evolved during cooling has maintained a heat gradient for a longer time and hence crystallization was more complete.

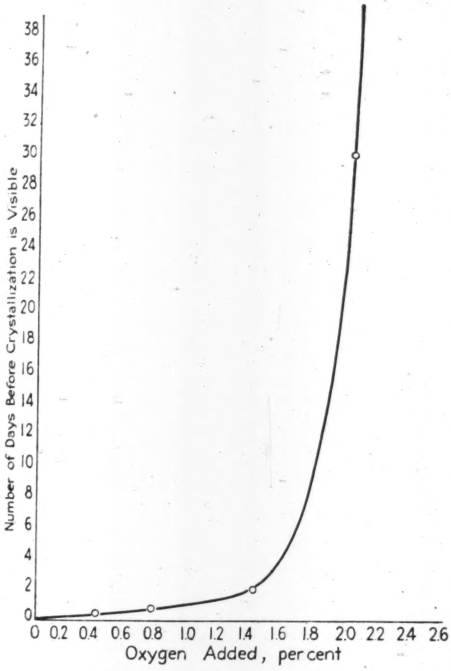

FIG. 7.—Effect of Temperature Gradients on Tube of Rosin.

Attempts have been made at times to prevent crystallization by the addition of various substances. Up to the present, these materials have not materially aided in solving the problem. Instances have been reported where certain treatments have tended to retard crystallization, but almost always the acid number has been reduced ten or twenty units by the treatment.

Considered from a theoretical standpoint, crystallization can be prevented by the addition of a material which is so strongly adsorbed that the crystal nuclei cannot form. Substances which are moderately absorbed only cause a change in crystal form. The effect of such on adsorption is shown in Fig. 8, which shows the effects of increasing amounts of water in the alcohol from which abietic acid was recrystallized. By noting the effect of other acidic and

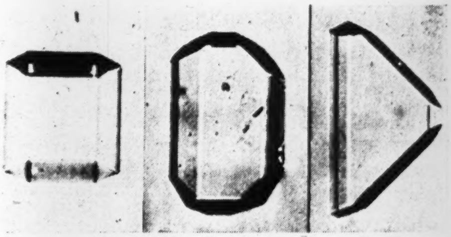

FIG. 8.—Effect of Solvents on Crystal Forms of Abietic Acid.

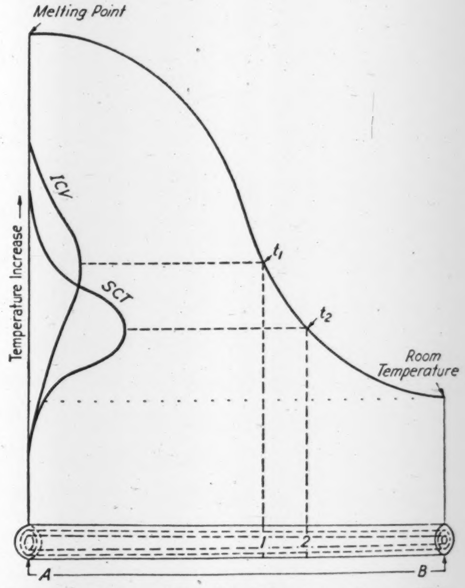

FIG. 9.—Effect of Oxidized Rosin on Crystallization Tendency of Rosin.

basic materials, we can determine whether anions or cations are more strongly adsorbed[11] by the abietic acid lattice.

It is known also that the tendency to supercool or form a glass is directly proportional to the number of hydroxyl groups in the molecule. Perhaps this is why oxidized rosin acts as a good crystallization preventive. Figure 9 shows data obtained on a grade I wood rosin containing various quantities of added oxidized rosin. Crystallization tests were carried out in an oven at 100 C. and the results showed quite conclusively that the presence of more than 1.5 per cent of oxidized material is sufficient to change enormously the results obtained. We mention this because when running oven crystallization tests for periods longer than 50 hr., sufficient oxygen is picked up by thin layers of sample to completely invalidate the results; hence, such oven tests should be carried out either with thick layers of sample, or preferably in an inert atmosphere.

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[11] Saylor, "Fifth Colloid Symposium Monograph," p. 49, The Chemical Catalog Co., Inc., New York City (1928).

DISCUSSION

Mr. H. E. Smith.[1]—The statement that rosin is a liquid and not a solid is interesting, yet in the cooling curves I notice that we have one characteristic of a solid. As a melted solid cools down there is a retardation in the rate of cooling at the point of solidification, where the energy of the melted liquid is released; and this continues for some time. With some materials—let us take very hot steel—after solidification we have what is known as the recalescence point, where there is a similar retardation of the cooling or even an increase in the temperature. In only a moderate light, you can see the steel brighten up; it becomes evidently hotter. That is due in steel to a rearrangement of the combinations of the ultimate constituents. I wonder whether there is something similar in rosin, and whether we could have any more details on that feature.

Mr. E. A. Georgi.[2]—I think the best analogy to that that we can make is simply to consider salt, common table salt, if you will, and water. On cooling, when you pass the saturation point, crystals of salt will come out. In the case of rosin the crystals of some rosin acid will come out. Now, when the temperature is raised, starting from a solution which contains the crystals—as that temperature is raised the crystals will gradually disappear and go back in solution, and if it is done slowly enough there will be no apparent change in the temperature of heating, or rather, in the rate of heating. Of course, if the sample is highly crystalline then there will be. There will be a hesitation in the temperature rise until the material is all melted, and then the temperature will go on up at a normal rate again. The two sets of cooling curves in my paper indicate that non-crystalline rosin had a continuous curve that comes down normally. When the sample of rosin crystallized, then one obtained a discontinuous change in the cooling curve. Of course, actually rosin is never a solid. At least, I don't like to consider it as a solid, even when crystalline. It is then simply a solution, plus crystals. In the case of metal, you probably never have an amorphous metal, unless you want to believe the theories of amorphous metal at the grain boundaries, etc. The analogy would have to be a pure rosin acid; it would crystallize completely. In regard to solid transformations, I have never found a solid transformation in rosin or any of the rosin acids.

The Chairman (*Mr. R. D. Bonney*[3]).—Mr. Georgi stated that crystalline rosin and ordinary rosin are the same, except for physical condition. I believe that in the literature there has been put forward evidence that crystalline rosin at a high temperature, say 400 F. or over, fails to react with lime and other materials in the way that ordinary rosin does. Now if the difference between ordinary rosin and crystalline rosin is merely in its physical condition, why should that affect the reaction of the molten material at high temperatures?

Mr. Georgi.—I do not believe it

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[2] Experiment Station, Hercules Powder Co., Wilmington, Del.

[3] Assistant Manager of Manufacturing, Congoleum-Nairn, Inc., Kearny, N. J.

does. I believe the reason for apparent non-reactivity is that a crystalline rosin, when heated to 400 F. in the presence of lime, does not react as fast as does liquid rosin and hence the rosin is usually not held at 400 F. long enough to allow all the crystals to melt, and then subsequently to react with the lime. Of course, the over-all picture will be of the rosin being less reactive but actually, if one holds the rosin at 400 F. long enough for all the crystals to melt, I think you will find that it will be just as reactive as any non-crystalline rosin.

STUDIES IN THE OXIDATION OF MINERAL TRANSFORMER OIL

By F. M. Clark[1]

Synopsis

Various tests have been suggested for determining the suitability of mineral transformer oil for commercial use. Among such tests have been the "life test" and the sludge accumulation test. The erratic results obtained with such testing procedures have been associated chiefly with the difficulty of exactly reproducing the chemical and mechanical test factors over the long periods of oxidation time involved. These defects appear to be eliminated by the oxidation test in which the oxidation of the oil is accelerated by the use of oxygen gas at 250 lb. per sq. in. pressure, the oxidation being carried out at 140 C. for a period of 24 hr. Sludge formation under such conditions of oxidation appears to be closely related to the formation of free organic acid bodies formed as a direct oxidation product and to involve no chemical factors not present in the oxidation and sludging of the oil under atmospheric conditions at lower temperature. Extreme oil oxidation under the conditions studied gives deviation from the normally expected linear relations. This deviation is accompanied by deep-seated chemical changes, the occurrence of which is manifested by the evolution of hydrogen and hydrocarbon gases and an oxidation which under some conditions may occur with extreme rapidity.

Accepting as a laboratory test procedure the oxidation of mineral transformer oil at 140 C., under 250 lb. per sq. in. of oxygen gas pressure, for a period of 24 hr., the sludge results obtained show a definite relation to the life test and sludge accumulation test results. The use of this high pressure test eliminates the long periods of oxidation normally associated with the life and accumulation type of sludge test with resulting increase in the reliability and ease of test duplication either in the same or different laboratories. The results of the high-pressure oxidation test are reproducible within a sludge value of approximately 0.003 per cent.

In determining the suitability of mineral transformer oil for commercial use, it is customary to examine the chemical stability of the material by means of an oxidation under suitably convenient conditions of temperature and time. A variety of methods have been selected for such a test procedure, the examination placing particular emphasis on the presence and amount of insoluble material (sludge) separating from the oil. In some instances, the examination has been extended to include acid formation in the oil. One such test is the "life test"[2] which determines the length of time at 120 C. for sludge to make its appearance under definitely prescribed conditions. Another such test is the sludge accumulation test[3] which determines the rate of sludge precipitation under conditions of test similar in most respects to those applying to the "life test" method.

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[1] Chemist, General Electric Co., Pittsfield, Mass.

[2] E. A. Snyder, "Method for Determining Sludging Properties of Transformer Oils," *Proceedings*, Am. Soc. Testing Mats., Vol. 23, Part I, p. 448 (1923).

The results obtained after a thorough investigation of each of these test methods have been generally unsatisfactory. Oxidation extending over the long periods of time necessary at 120 C. to evaluate properly the sludging characteristics of the oil have yielded widely erratic test values which become even more scattered when a catalyst such as copper is used in order to accelerate the oxidation effects.

One of the marked defects generally associated with the laboratory examination of mineral transformer oil is the long period of oxidation time usually prescribed. In an examination extending over weeks and months, slight variations in the oxidizing conditions are of marked effect on the accumulated reaction results. Such conditions include the maintenance of a non-varying temperature, a non-varying air supply, freedom from contamination of all types and, in the case of a copper catalyst, exact reproduction of the metal surface condition from test to test. It is rarely if ever possible to meet these requirements completely. Still further complications are introduced by the evaporation of the oil itself during the oxidation run. In the usual sludge accumulation oxidation test involving a period of 55 days at 120 C., the loss of oil by evaporation may range from 20 to 33 per cent of that originally present. Such oil evaporation, coupled with the accompanying volatilization of many of the oil oxidation products, severely alters the oxidation and sludge test results.

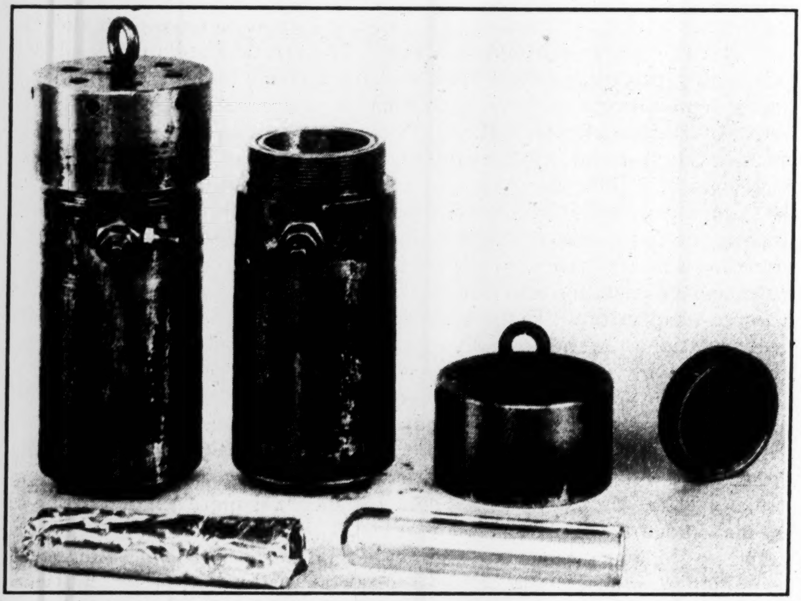

FIG. 1.—Apparatus for Testing Oxidation Characteristics of Transformer Oils at High Oxygen Pressure.

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[3] F. M. Clark and E. A. Snyder, "Testing for Sludge Formation in Mineral Transformer Oil," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 568 (1936).

The present paper presents an analysis of the oxidation of mineral transformer oil under such conditions that evaporation losses are eliminated and each succeeding stage of oxidation is subject to the accelerating or retarding effects of the products from earlier oxidation. This is accomplished by means of oxidation in closed containers at higher than atmospheric pressure. Under these conditions with but little increase in oxidation temperature, the chemical changes are accelerated to the extent that periods of oxidation running into weeks and months are eliminated with correspondingly greater ease in the reproduction and maintenance of the various oxidation factors.

Apparatus Used:

The oil oxidation apparatus is illustrated in Fig. 1. The oxidation cell is of stainless steel so constructed as to handle safely the oxidation of mineral oil under the gas pressure used, which is normally of the order of 250 lb. per sq. in. The oxidation chamber is cylindrical in shape and approximately $7\frac{1}{2}$ in. long and $2\frac{1}{2}$ in. in diameter. The oil during oxidation is placed in a Pyrex glass tube 6 in. long and $1\frac{1}{8}$ in. in diameter. Approximately 50 ml. of oil are used, weighed carefully, with the usual gravimetric analytical precautions. When a copper catalyst is used, 12 in. of 0.040-in. copper wire is cleaned mechanically to produce a fresh surface, washed with chemically pure ethyl ether, dried and wound on a 1-in. mandrel, the over-all length of the spiral so produced being $\frac{1}{2}$ in. In all such tests, the copper catalyst is always completely immersed in the mineral oil.

Oil Used:

Throughout this report the oil used has been a typical American transformer oil, the characteristics of which are given in Table I.

While the oxidation principles described are applicable to all types of oil, modification is necessary as one changes from one kind of oil to another. A highly refined, water-white, paraffinic oil, for example, will oxidize with extreme rapidity under some conditions of test with the higher oxygen pressures.

TABLE I.—THE CHEMICAL AND PHYSICAL CHARACTERISTICS OF THE MINERAL TRANSFORMER OIL EXAMINED.

A.S.T.M. color number	$\frac{1}{2}$
Specific gravity (15.5/15.5 C.)	0.885
Saybolt Universal viscosity (37.8 C.)	58 sec.
Refractive index (25 C.)	1.4865
Dielectric strength (25 C.)	30 kv.
Total sulfur content	0.10 per cent
Free sulfur content	0
Neutralization number	less than 0.01 mg of KOH per g.

Test Methods:

Throughout this report the oil test methods used have followed the procedures outlined by this Society.[4] Sludge precipitation has been determined in accordance with the method already described for the sludge accumulation test.[3] This comprises the precipitation of the sludge with naphtha, after which it is washed with naphtha, dried and

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[4] The following Society testing procedures have been followed:

Tentative Method of Test for Neutralization Number of Petroleum Products and Lubricants (D 188-27 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 27, Part I, p. 927 (1927); also 1937 Book of A.S.T.M. Tentative Standards, p. 737.

Standard Method of Test for Gravity of Petroleum and Petroleum Products by Means of the Hydrometer (D 287-37), 1937 Supplement to Book of A.S.T.M. Standards, p. 121.

Tentative Method of Test for Sulfur in Petroleum Oils by Lamp Method (D 90-34 T), Proceedings, Am. Soc. Testing Mats., Vol. 34, Part I, p. 911 (1934); also 1937 Book of A.S.T.M. Tentative Standards, p. 751.

Standard Methods of Testing Electrical Insulating Oils (D 117-36), 1936 Book of A.S.T.M. Standards, Part II p. 1167.

Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter (D 88-36), 1936 Book of A.S.T.M. Standards, Part II, p. 982.

Tentative Method of Test for Saponification Number of Electrical Insulating Oils (Modified Baader Method) (D 438-36 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 988 (1936); also 1937 Book of A.S.T.M. Tentative Standards, p. 1113.

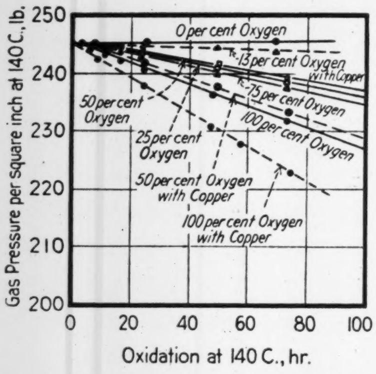

FIG. 2.—Gas Absorption by Mineral Transformer Oil as a Function of the Duration of Oxidation at 140 C. The oxidizing gas mixture consists of oxygen and nitrogen as stated.

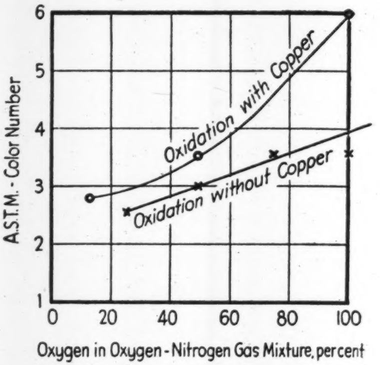

FIG. 3.—Color Change in Mineral Transformer Oil During 24 hr. of Oxidation at 140 C., as Affected by the Composition of the Oxygen-Nitrogen Gas Mixture Applied at a Pressure of 250 lb. per sq. in.

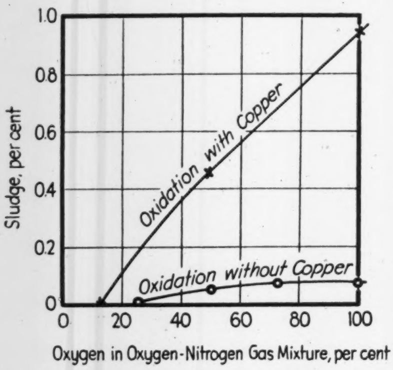

FIG. 4.—Sludge Formation in Mineral Transformer Oil During 24 hr. of Oxidation at 140 C., as Affected by the Composition of the Oxygen-Nitrogen Gas Mixture Applied at a Pressure of 250 lb. per sq. in.

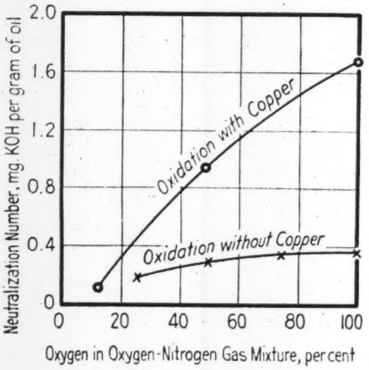

FIG. 5.—The Change in Neutralization Value of Mineral Transformer Oil During 24 hr. of Oxidation at 140 C., as Affected by the Composition of the Oxygen-Nitrogen Gas Mixture Applied at a Pressure of 250 lb. per sq. in.

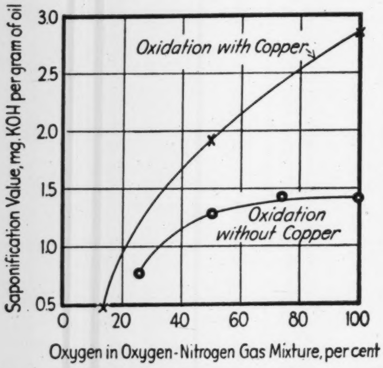

FIG. 6.—The Change in Saponification Value of Mineral Transformer Oil During 24 hr. of Oxidation at 140 C. as Affected by the Composition of the Oxygen-Nitrogen Gas Mixture Applied at a Pressure of 250 lb. per sq. in.

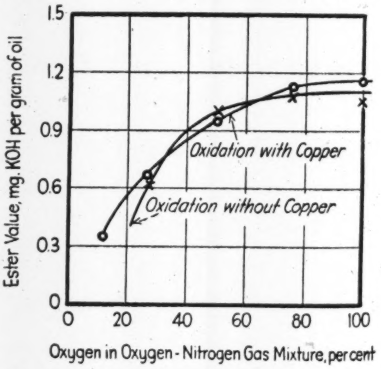

FIG. 7.—The Change in Ester Value of Mineral Transformer Oil During 24 hr. of Oxidation at 140 C. as Affected by the Composition of the Oxygen-Nitrogen Gas Mixture Applied at a Pressure of 250 lb. per sq. in.

weighed, the amount present being expressed in percentage by weight of the original oil used.

The ester value is indicative of acid bodies removed by reaction with metals or organic hydroxyl-containing compounds. This value is obtained as the difference between the saponification value and the neutralization number, both expressed in terms of milligrams of potassium hydroxide per gram of oil.

The water soluble acidity value is the neutralization number of the water extract obtained by exhaustively washing the oxidized oil with distilled water, the value being expressed as the milligrams of potassium hydroxide per gram of the oxidized oil extracted.

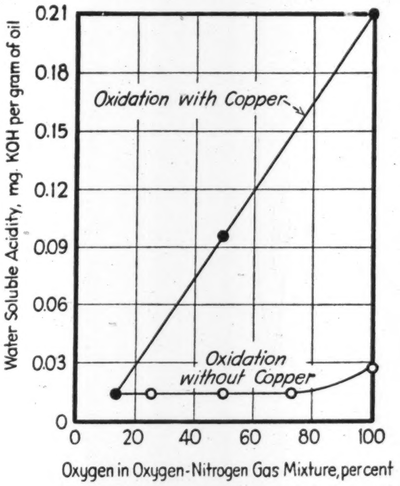

FIG. 8.—The Change in Water-Soluble Acidity of Mineral Transformer Oil During 24 hr. of Oxidation at 140 C., as Affected by the Composition of the Oxygen-Nitrogen Gas Mixture Applied at a Pressure of 250 lb. per sq. in.

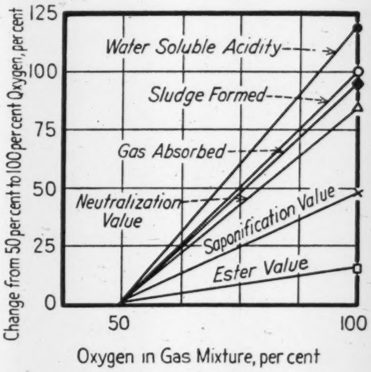

FIG. 10.—The Relation Between Gas Absorption and the Oxidation of Mineral Transformer Oil Heated with Copper at 140 C. for 24 hr. Under a Gas Pressure of 250 lb. per sq. in.

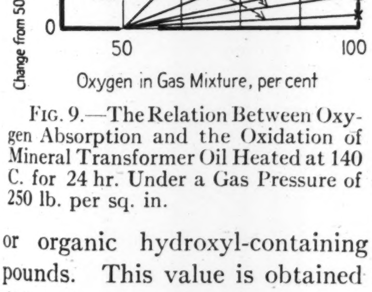

FIG. 9.—The Relation Between Oxygen Absorption and the Oxidation of Mineral Transformer Oil Heated at 140 C. for 24 hr. Under a Gas Pressure of 250 lb. per sq. in.

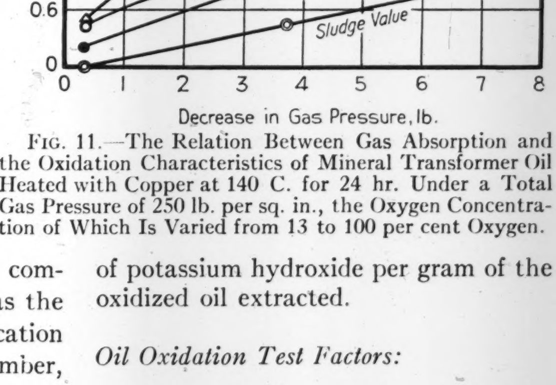

FIG. 11.—The Relation Between Gas Absorption and the Oxidation Characteristics of Mineral Transformer Oil Heated with Copper at 140 C. for 24 hr. Under a Total Gas Pressure of 250 lb. per sq. in., the Oxygen Concentration of Which Is Varied from 13 to 100 per cent Oxygen.

Oil Oxidation Test Factors:

In the examination of the oxidation characteristics of mineral transformer oil, careful study must be made of the effects of oxygen concentration, temperature change, variation in the time factor and the oxygen-containing gas pressure. The adoption of any factor so severe as to cause change in the chemical nature of the composition aside from oxidation, even though these changes be directly dependent on oxidation must be avoided. The immediately following paragraphs therefore concern the effect of each factor, separately considered, on the oxidation behavior of the oil under test.

Effect of Oxygen Concentration:

Oxidized at 140 C. under 250-lb. gas pressure, the gas absorption is a linear function of the oxidation time. This is illustrated in Fig. 2. The linear rate of oxygen absorption holds whether a copper catalyst be present or not. The linear relation of gas absorption with time of oxidation is taken as an indication of the absence of chemical abnormality in the oxidation reaction for each of the oxygen concentrations investigated.

Figures 3 to 8 illustrate, respectively, the change in oil color, sludge formation, neutralization number, saponification, ester and water-soluble acid formation produced by oxidation under 245 lb. (initial) gas pressure at 140 C. for 24 hr. The effect of oxygen concentration in the gas mixture is shown. Figure 9, based on the data of Figs. 3 to 8, illustrates the change in the various oil properties when the oxygen concentration of the reacting gas is increased from 50 per cent to 100 per cent. Although the amount of gas absorbed increases in direct proportion to the oxygen content of the reacting gas, the only oil property which changes in corresponding amount is the total water-soluble acidity. The rate of oxygen absorption as indicated by the drop in gas pressure during the oil oxidation in the absence of copper is, therefore, not closely related to sludge formation and does not appear to be a proper gage for its evaluation.

In the presence of copper, however, oxygen absorption does appear to have significance. The oil oxidation changes are more pronounced and a definite relation appears between the amount of oxygen absorbed as indicated by gas pressure change and such important transformer oil properties as neutralization value and sludge formation. Figure 10 shows the percentage change in the properties of the oil and gas absorption in relation to the concentration of oxygen in the gas mixture used. All data are based on Figs. 3 to 8. It is to be observed that the water-soluble acidity, sludge formation and neutralization value of the oil, as well as the amount of gas absorbed, change in approximately a direct relation to the change in oxygen concentration from 50 per cent to 100 per cent. Figure 11 gives a more complete summary of the data of Figs. 3 to 8 with respect to the oxidation in the presence of copper. Figure 11 shows the change in oil test value as related to the gas absorption as the oxygen concentration of the gas is varied from 13 per cent to 100 per cent of the reacting gas mixture. The linear relation between gas absorption and the oil properties of neutralization and sludge formation appears to be of fundamental importance. The saponification and ester-forming properties of the oil are non-linearly related to the gas absorption. Complete discussion of the significance of the various oil characteristics and their relation to sludge formation is reserved for later paragraphs.

Gases Formed During Oil Oxidation:

In the commercial use of mineral transformer oil, chemical change appears confined to an oxidation reaction. No evidence of molecular "cracking" is obtained. Laboratory examination of the oil in order to gage its suitability for practical application must, therefore, be limited to a study of the simple oxidation and sludging reactions. Molecular changes of other types must be avoided. Examination of the gases evolved when mineral transformer oil is oxidized at 140 C. under 250 lb. per sq. in. oxygen pressure indicates that unless the oxidation be extended beyond 100 hr., only oxidation gases such as carbon dioxide and carbon monoxide are obtained. Carried as far as 168 hr., however, clear evidence of molecular cracking is obtained as indicated by the presence of saturated and unsaturated gaseous hydrocarbons and hydrogen. Typical gas analysis taken after 100 and 168 hr. of oil oxidation is given in Table II. The presence of metallic copper in the oil during the oxidation has little effect on the analytical test results. The data of Table II do not include the oil-dissolved gases.

Influence of Oxygen Pressure, Temperature, and Time on the Oxidation and Sludging Reactions:

When changes are made in those factors which determine the rate of oxidation in mineral transformer oil under normal conditions of usage, care must be exercised that the increase in oxidation rate is not obtained by the introduction of reactions normally absent. An oxidation temperature of 110 to 115 C. has been suggested as the maximum allowable. Investigation of American transformer oil, however, has shown that at temperatures as high as 130 C. no changes are introduced that would tend to vitiate the significance of the chemical results obtained. In view of the data of Fig. 2 and the gas analysis of Table II, it is concluded that at 140 C., under 250 lb. per sq. in. oxygen pressure, the oxidation can be safely extended to about 100 hr. The data of this section constitute a further survey of the chemical changes occurring as the time, temperature, and oxygen pressure are successively changed in order to obtain widely varying rates of oxidation.

Considering the problem entirely from the standpoint of sludge formation—one of the principal properties which determines the suitability of mineral transformer oil—changes in the oxidation temperature, time, and oxygen pressure invariably produce a linear effect on the sludge formed. This is illustrated in the data of Figs. 12, 13 and 14 which describe, respectively, the effect of oxygen gas pressure change on the oil oxidation at 140 C. for a period of 24 hr., the effect of temperature change on the oxidation at 250 lb. oxygen pressure for 24 hr., and the effect of time on the oxidation at 140 C., 250 lb. oxygen pressure.

TABLE II.—GASES FORMED DURING THE OXIDATION OF MINERAL TRANSFORMER OIL AT 140 C. UNDER 250 LB. PER SQ. IN. OXYGEN PRESSURE.

	NO OXIDATION	24-HR. OXIDATION	100-HR. OXIDATION	168-HR. OXIDATION
Oxygen, per cent	100	99.4	96.8	78
Carbon dioxide, per cent	0	0.3	2.0	15.5
Carbon monoxide, per cent	0	0.3	1.0	2.8
Hydrogen, per cent	0	0	0	3.0
Saturated hydrocarbons, per cent	0	0	0	0.5
Unsaturated hydrocarbons, per cent	0	0	0.2	0.2

The data of Figs. 12, 13 and 14 show no abnormality indicative of the introduction of other than an oxidation reaction within the variation of test factors investigated. When, however, the oxidation is carried beyond 100 hr. at 140 C. under 250 lb. oxygen pressure, a depar-

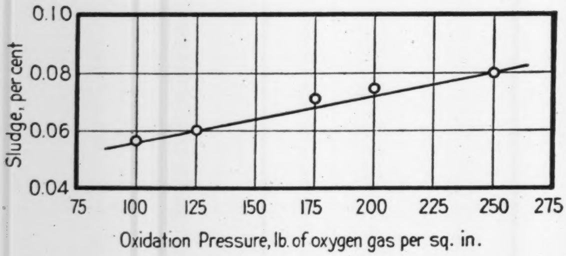

FIG. 12.—Sludge Formation in Mineral Transformer Oil as Affected by Oxygen Gas Pressure During a 24-hr. Oxidation at 140 C.

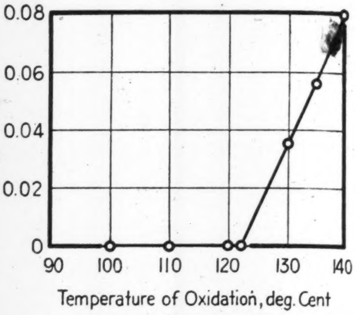

FIG. 13.—Sludge Formation in Mineral Transformer Oil as Affected by the Temperature During a 24-hr. Oxidation at 250 lb. per sq. in Oxygen Gas Pressure.

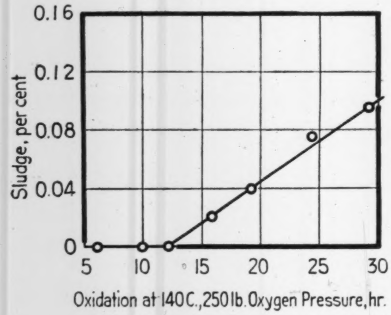

FIG. 14.—Sludge Formation in Mineral Transformer Oil as Affected by the Duration of Oxidation at 140 C. Under an Oxygen Pressure of 250 lb. per sq. in.

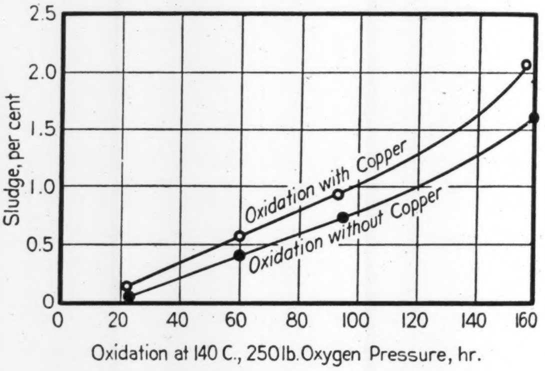

FIG. 15.—Sludge Formation in Mineral Transformer Oil as Affected by Extended Periods of Oxidation at 140 C. Under 250 lb. per sq. in. of Oxygen Gas Pressure.

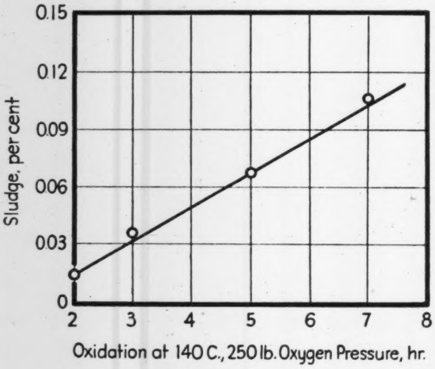

FIG. 16.—Sludge Formation in Mineral Transformer Oil in the Presence of Copper as Affected by the Duration of Oxidation at 140 C. Under an Oxygen Pressure of 250 lb. per sq. in.

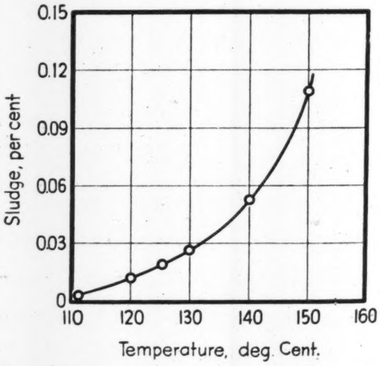

FIG. 17.—Sludge Formation in Mineral Transformer Oil in the Presence of Copper as Affected by the Temperature During a 7-hr. Oxidation at 250 lb. per sq. in. Oxygen Pressure.

ture from the linear relation of sludge formation to duration of oxidation is obtained. As illustrated in Fig. 15, this non-linear relation is obtained irrespective of the presence or absence of copper. The departure from a linear relation is accepted as indicating an abnormality in the oxidation and sludging reactions, which in view of the data of Table II is assumed to be related to deep-seated chemical changes associated with the generation of hydrogen and hydrocarbon gases.

With copper present, substantially similar results are obtained as when the oxidation is carried out in the absence of copper. Linear relationships are observed between the sludge value and the duration of the oxidation. The temperature effect on sludge formation in the presence of copper is linear between 110 and 130 C. For higher temperature a non-linear relation results in which the sludge accumulates at a rate more rapid than the temperature increase. These effects are illustrated in Figs. 16 and 17.

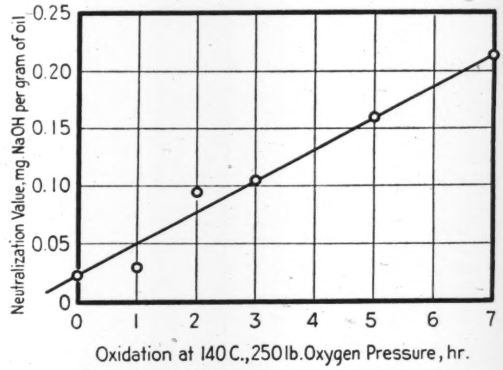

FIG. 18.—The Neutralization Value of Mineral Transformer Oil as Affected by the Duration of the Oxidation in the Presence of Copper at 140 C. Under 250 lb. per sq. in. Oxygen Pressure.

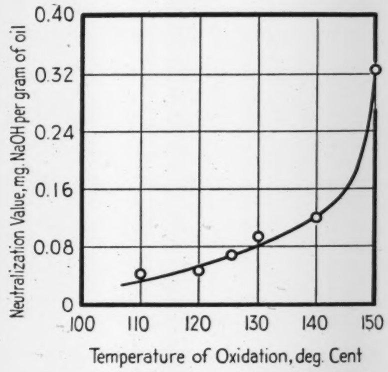

FIG. 19.—The Neutralization Value of Mineral Transformer Oil as Affected by the Temperature of Oxidation in the Presence of Copper at 250 lb. per sq. in. Oxygen Pressure for 7 hr.

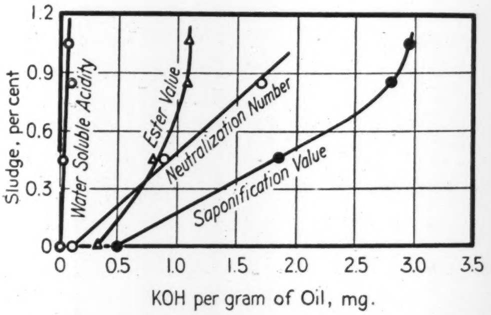

FIG. 20.—The Relation of Sludge Formation and the Oxidation Characteristics of Mineral Oil Produced by High Temperature and High Oxygen Pressure Oxidation in the Presence of Copper.

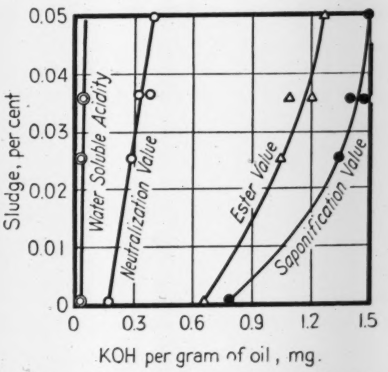

FIG. 21.—The Relation of Sludge Formation and the Oxidation Characteristics of Mineral Oil Produced by High Temperature and High Oxygen Pressure Oxidation in the Absence of Copper.

Figure 17 again demonstrates the deep-seated chemical changes which accompany extreme oxidation, changes which, unless carefully evaluated, may vitiate the importance of laboratory examinations in gaging the suitability of mineral oil for transformer use. The oil neutralization value reflects the sludge studies. Only with extreme oxidation does a non-linear relationship result. This is illustrated in Figs. 18 and 19.

Relation of Sludge Formation to Oxidized Oil Characteristics:

In the oxidation of mineral transformer oil, a series of oxidation reactions is envisaged, some of which have little if any relation to the actual sludge precipitation. Sludge formation is usually looked upon as a secondary reaction involving condensation or polymerization of previously formed oxidation products. It therefore becomes of importance to determine which oxidation products are directly related to the later occurring sludge formation reaction. Color change is, of course, indicative of fundamental changes affecting the chemical nature of at least some of the chemical compounds present. Oil color from a practical standpoint, however, is not a reliable standard because of the contaminating influence of oil-soluble materials dissolved in the handling or use of the oil in transformer service. The neutralization value, saponification number and ester value are chemical changes susceptible to reliable measurement and are worthy of inspection in their relation to sludge formation. The similarity in the neutralization characteristics and the sludge-forming properties of the oil examined in these studies suggests an interrelation. In like manner, the lack of similarity in the saponification and ester-forming properties of the oil as compared to sludge formation is indicative of chemical changes not closely related to the latter.

Figures 20 and 21 summarize the general relation between the various oxidized oil characteristics and sludge formation. In some instances, especially in the absence of copper, the relation of ester value and saponification has given a concave downward type of curve, but in no instance has a linear relation with sludge value been obtained. With or without copper, the neutralization value and water-soluble acid value have always changed in a linear relation to the sludge value. Sludge formation appears, therefore, directly related to the formation of organic acids during the oxidation.

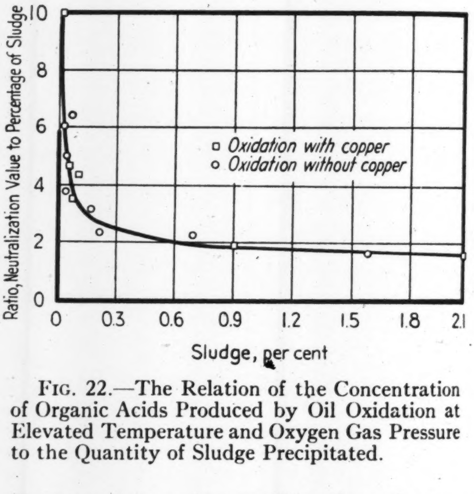

Fig. 22.—The Relation of the Concentration of Organic Acids Produced by Oil Oxidation at Elevated Temperature and Oxygen Gas Pressure to the Quantity of Sludge Precipitated.

Ester value and saponification characteristics clearly appear to be the result of reaction between oil oxidation products important in their effects only through the resultant removal of the more important free acid bodies.

The formation of free organic acids occurs in the early stages of oil oxidation. In a well-refined transformer oil of the type under investigation, the separation of sludge under the conditions of oxidation described does not begin until the oil neutralization value has become well marked. With the accumulation of sludge, the ratio of the neutralization value to sludge formation rapidly decreases to an approximately constant value of 1.75, which appears to be closely maintained within wide variations of the sludge and neutralization value. The relationship is illustrated in Fig. 22. For total amounts of sludge less than approximately 0.5 per cent by weight, the ratio of the neutralization value to sludge value changes rapidly with small changes in the amount of sludge present. It is in this range of sludge values that most of the empirically established tests have been concerned. It is suggested that much of the confusion and erratic nature of the test results might be eliminated were the tests so altered as to produce sludge in excess of 0.5 per cent and preferably to about 1 per cent.

Data summarized in Fig. 22 clearly suggest that sludge formation in a typical American transformer oil involves a secondary reaction, one reactant of which is the free organic acid component of the oil formed as a result of a primary oxidation. The formation of organic esters has little relation to sludge precipitation except in so far as such formation diminishes the free organic acid component of the oil. The practical evaluation of a transformer oil during service use which includes the determination of the neutralization value of the oil appears, therefore, to be well grounded.

A Laboratory Test for the Gaging of Sludge Formation in Mineral Transformer Oil:

For the proper gaging of sludge formation in mineral transformer oil, the test procedure to be of real value from the standpoint of reproducibility in one or many laboratories should preferably be of short duration in order to reduce or eliminate the difficulties of mechanical control. The conditions used to reduce the duration of test, however, must not be exaggerated to the extent that chemical abnormalities are introduced which increase the difficulties in the translation of academic laboratory findings into terms of practical use. The data already presented suggest a laboratory test procedure for gaging sludge formation in new and unused mineral transformer oil. Such a test, in order to avoid the abnormalities of extreme oxidation, should involve a temperature not higher than about 150 C. and extend over a period of time not greater than 100 hr. The oxygen gas pressure applied is conveniently accepted at 250 lb. per sq. in. With previously oxidized transformer oil, however, the extent of oxidation to which it can be submitted, without deep-seated chemical changes involving factors other than oxidation, is reduced in proportion to the degree of the previous oxidation. These deep-seated chemical oil changes must be avoided if the sludging property of the oil is to be accurately and safely determined. The test procedure accepted for further investigation therefore comprises an oil oxidation under the following conditions:

Apparatus.......	as illustrated in Fig. 1.
Temperature.....	140 C.
Oxidizing gas....	dry oxygen substantially 100 per cent pure.
Oxygen pressure..	250 lb. per sq. in. at 140 C.
Duration of oxidation........	24 hr.
Volume of oil....	50 ml.
Oil container.....	as described for Fig. 1.
Preparation of copper catalyst if used........	0.040-in. copper wire, mechanically cleaned and washed and wound on a spiral as described in connection with Fig. 1.

The sludge formed is determined in accordance with the following procedure:

After the prescribed interval of oxidation, the tube containing the oil under examination is removed and allowed to stand for 24 hr. at room temperature, protected from light and possible contamination. The oil is then diluted with an amount of naphtha equal to 75 to 80 per cent of the oil volume. The naphtha used meets the requirements for naphtha given in the A.S.T.M. Standard Method of Test for Precipitation Number of Lubricating Oils (D 91 - 35).[5] The naphtha-oil solution is then centrifuged at 1800 r.p.m. for 20 min. The supernatant liquid is carefully decanted and the sludge washed at least twice with naphtha or until no stain is produced on filter paper by a drop of the naphtha washing. The tube with its sludge, after having been properly washed, is dried in a 100 C. oven for 3 hr., the tube being inclined at a 10-deg. angle from horizontal. The dried tube and sludge, after having been cooled in a desiccator, is weighed with the usual gravimetric analytical precautions, freed from sludge by cleaning with acetone or other suitable volatile solvent or cleaning solution, dried at 100 C. and reweighed in order to obtain the total weight of the sludge formed. The weight of sludge is expressed in percentage by weight of the original oil.

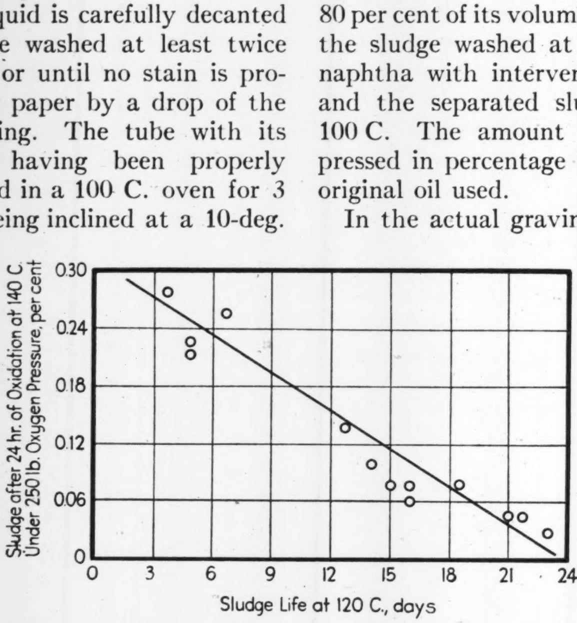

FIG. 23.—The Relation of the Life Test Value in Mineral Transformer Oil to the Quantity of Sludge Precipitated as a Result of a 24-hr. Oxidation at 140 C. Under 250 lb. per sq. in. Oxygen Gas Pressure.

When copper is used to accelerate sludge formation, the examination of the oxidized oil for sludge content is exactly as followed for the no-copper test, modified as is necessary due to the presence of copper. After having been cooled to room temperature, and held for 24 hr., the copper spiral is removed from the oxidized oil and washed with a spray of the specified naphtha. The oil is then diluted with naphtha to 75 to 80 per cent of its volume and centrifuged, the sludge washed at least twice with naphtha with intervening centrifuging, and the separated sludge is dried at 100 C. The amount of sludge is expressed in percentage by weight of the original oil used.

In the actual gravimetric determina-

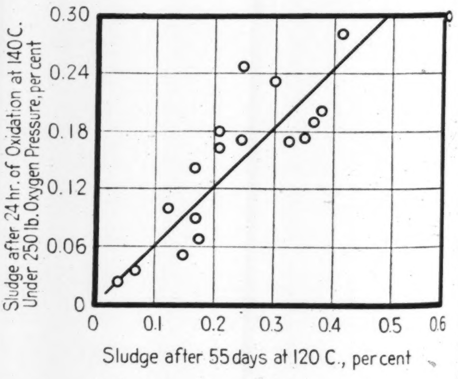

FIG. 24.—The Relation of the Sludge Accumulation Test Value to the Amount of Sludge Precipitated from Mineral Transformer Oil as a Result of a 24-hr. Oxidation at 140 C. Under 250 lb. per sq. in. Oxygen Gas Pressure.

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[5] 1936 Book of A.S.T.M. Standards, Part II, p. 944.

tion of the sludge formed, the use of a Gooch or sintered-glass crucible in place of the procedure described has produced no important variation.

The procedure as outlined has been found to give results reproducible in the same laboratory with a maximum variation in the sludge value of 0.003 per cent. To obtain such reproducibility, it is important to note that, whether copper be present or absent, the important test factor is a properly controlled test temperature. A variation of 1 deg. Cent. in the average temperature produces a change of 0.003 to 0.005 per cent in the sludge value.

The pressure and time factors are easily controlled. Of these, change in gas pressure from 250 lb. per sq. in. produces only 0.001 to 0.002 per cent change in sludge value for each 10 lb. pressure variation. The sludge formed is increased by an amount ranging from 0.002 to 0.009 per cent for each 30-min. increase in the standard 24-hr. value, the amount of change depending on the presence or absence of copper.

In carrying out the standard test as outlined, it is specified that the period of oxidation shall be 24 hr., the average temperature 140 C. with a maximum variation of ± 1 deg. Cent., and an initial gas pressure at 140 C., of 250 lb. per sq. in. of oxygen with a deviation not exceeding 5 lb. per sq. in. With such testing routine, check tests in the same laboratory are obtained with a difference not exceeding 0.003 per cent. Within the limits of experience so far obtained, check tests on the same new and unused oil in different laboratories have agreed within a variation of 0.010 per cent.

Correlation of High-Pressure Oxidation and Suggested Sludge Testing Methods:

Accepting as a testing procedure the determination of the amount of sludge formed in a mineral transformer oil after 24 hr. of oxidation at 140 C. under 250 lb. oxygen pressure, Figs. 23 and 24 illustrate the correlation in test results with the life test value and the sludge produced in accordance with the sludge accumulation procedure after 55 days at 120 C. All test results were obtained without the use of a copper catalyst. The oil in these tests was of the type described in Table I in various stages of purity.

From the variation in test results recognized as inherent in the "life test" and sludge-accumulation test procedure, a close correlation with any other type of test cannot be expected. In a general sense, however, Figs. 23 and 24 demonstrate a relation sufficiently pronounced to indicate that the analytical advantages of the high-pressure oxidation are obtained merely through the acceleration of chemical reactions normally operative in the lower temperature-lower pressure tests heretofore investigated.

DISCUSSION

Mr. Herman Halperin[1] (*presented in written form*).—This new test method evolved by Mr. Clark has the following desirable features: short duration, use of copper catalyst, closed system preventing escape of volatile oxidation products which may affect the course of oxidation. The test is superior to the Snyder life test in all these points. It is superior to the Baader test due to the last point.

The temperature of the new test is very high, 140 C. as compared with 120 C. for the Snyder test, 95 C. for the Baader test and 100 C. for the test evolved by H. W. L. Bruckman. Several investigators claim that the type of oil oxidation becomes essentially different from normal service oxidation if the oxidation temperature exceeds 120 C. As to the results in Table II of the paper, which is shown in support of the assumption that the test at 140 C. does not change the type of oxidation, it is quite conceivable that, especially in the initial stages of test, liberated hydrogen and unsaturates could combine with the oxygen to form water and peroxides and acids, respectively. The absence of hydrogen and gaseous hydrocarbons in the gas analysis appears to us, therefore, no reliable indication that break-up of the molecules has not occurred. As to the linear rate of oxidation found, both the research at Massachusetts Institute of Technology and Dornte's work show that this is not a general characteristic of oil but that some oils show an accelerated rate of oxidation (autocatalytic) while others show a retarded (inhibited) rate of oxidation.

Regardless of these arguments, the deciding factors on the value of an aging test are: (*a*) reproducibility within a laboratory, (*b*) reproducibility between laboratories, and (*c*) correlation with service performance. Mr. Clark gives information on only the first of these requirements and a restricted statement on the second.

Mr. Clark states that, while applicable in principle to all oils, the method needs modification "as one changes from one kind of oil to another." This limitation appears to us a serious objection because for comparative information all oils should be subjected to the same test and the test method should not be adjusted according to the judgment of the operator. It is not stated what modifications are necessary, but they are probably in temperature, pressure, or both. It is also not stated on what basis the operator should decide what modifications are necessary.

Summarizing, it can be said: A satisfactory stability test for transformer oil is an urgent need. Mr. Clark's test has some desirable features. Its reliability and value in predicting the service performance of oils has to be still proved by further investigations and experience. In this connection, it should be kept in mind that other methods also have seemed promising and later developed deficiencies in more general application. Mr. Clark's efforts in trying to develop an improved life test should be welcomed and supported.

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[1] Assistant Equipment and Research Engineer, Engineering Department, Commonwealth Edison Co., Chicago, Ill.

Mr. Clark did not give any limits to distinguish between good and bad oils in his test. He made his investigations on only one good oil. In this connection it should be noted that sometimes test methods give good reproducibility for good oils but not for inferior or used oils.

MR. S. J. ROSCH.[2]—If anyone else but Mr. Clark had presented this paper, we would have paid no attention to it because the data presented represents work on one oil only, and therefore must be inconclusive. However, knowing the high caliber of work usually performed by Mr. Clark, I feel that, although the data show the results of tests presumably on one oil only, he undoubtedly must have worked with a number of oils. As a matter of fact, during the presentation of his paper he admitted that he had investigated some water-white oils. I would therefore like to ask Mr. Clark whether his investigations also include some work on oils used in the impregnation of paper cables. It would be of real help to know that the same type of correlation as shown in the paper, was also applicable to these cable oils.

With regards to the type of bomb used by Mr. Clark, it may be pointed out that similar work was carried out by another laboratory but not using a stainless steel bomb. As a result of the high temperatures, oxidation caused severe corrosion of the bomb, so that in carrying on this type of work considerable attention must be paid to the nature of the metal used for the bomb.

With regards to the temperature of 140 C. selected by Mr. Clark, it is our feeling that 130 C. would not only be preferable, but would undoubtedly give the same type of results. We mention 130 C. because above this temperature, a form of deterioration may occur in the oil which may be misleading in so far as the original aim of the experiment is concerned. In other words, there is the possibility of breakdown of the structure due to possible cracking of the oil above 130 C. Mr. Clark may not agree with this but it is our feeling that some oils may show up very poorly at 140 C. but might show up fairly good, or exceptionally good, at 130 C. In view of the fact that Mr. Clark also carried on some of his investigation at 130 C., we would appreciate his views on the relative merits of carrying on this test at either of the two temperatures.

At the Massachusetts Institute of Technology, there is a special committee carrying on a similar investigation on the properties of insulating oils, and they have been able to obtain fairly close correlation between power factor measurements and sludge formation. They have also been able to tie this in with changes in the color of the oil. We would appreciate it if Mr. Clark would touch on this phase of the subject and indicate whether he has attempted to obtain any correlation between power factor values, or other electrical characteristics, and change in color or sludge formation.

MR. E. A. SNYDER.[3]—As chairman of Subcommittee IV on Insulating Mineral Oils of the Society's Committee D-9 on Electrical Insulating Materials, I simply want to say that for many years we have been trying to fit facts with theory in developing a transformer oil test. Unfortunately, over a period of years we seem to have used, without complete success, all the theories we had. They simply did not fit all the facts.

Over many years we have really been trying to correlate actual transformer operation with some kind of test on the oil that would show the expected life of the oil. So far all I can say for that work is that we have made steady progress. Mr. Clark now presents a new test, and I am glad that it has been brought out, not that I did not know about it before, but it had not, up to date, been in a state of development where Mr. Clark felt it was ready to present to this Society. Now that it has been presented with the data that he has shown in the paper, I can assure you that Subcommittee IV will be glad to take it up and study it.

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[2] Manager, Insulated Products Development, Anaconda Wire and Cable Co., Hastings-on-Hudson, N. Y.

[3] Insulating Oil Sales, Socony-Vacuum Oil Co., Inc., New York City.

You, who have worked with us in Subcommittee IV know the troubles we have experienced in trying to find out how to evaluate an insulating oil. I have spent many years with the Socony-Vacuum Co. and many years before that with the General Electric Co. studying insulating oils, and from my own experience I would suggest that to determine, for example, which of two oils is the better, you use all the tests that have been proposed, study your oils, evaluate them and then from the test results plus the light of your experience in operation, try to determine which of the two is the better. Then when you have selected one, put it into service and if service experience bears you out, your guess has been good. We have, in our own company, in the last four or five years, put in operation some thirty or more transformers in which we have tried various kinds of oils and we have been trying to correlate theory and fact, and repeatedly we have been brought right back to the fundamental thought that actual operation is the final criterion of insulating oil fitness. I do not say that this is the final answer and, as chairman of Subcommittee IV, I want to say that any addition or any suggestion to the committee for further study will be welcomed, because we really want to solve this problem.

THE CHAIRMAN (*Mr. H. C. Mougey*[4]).—I am really very much interested in the effect of catalysts and should like to ask Mr. Clark why the tests were made in glass. My reason for asking is that in our use of oils in automobiles we think the effect of the catalyst that the oil may pick up is of such great importance.

MR. F. M. CLARK[5] (*author's closure by letter*).—In general, the criticism expressed in the discussion of my paper involves the following factors:

1. The test temperature (140 C.) is too high, for there is a critical temperature at about 120 C. which must not be exceeded.
2. The linear rate of oxidation does not universally apply to all oils.
3. It has not been shown that the test procedure correlates with service practice.
4. The test procedure must be modified as the chemical nature of the oil tested is changed.
5. Only one type of oil is described.
6. Is it agreed that there is a relation between oil color, sludge formation and oil power factor?

Most of the discussion appears to ignore the fundamental object of the paper—the development of a *short time* test for gaging sludge formation in transformer oil. Obviously to accomplish this result, exaggeration in the test conditions is necessary. With the time factor fixed as of short duration, the only remaining factors which can be "exaggerated" are oxygen concentration and temperature. This test involves exaggeration of both. It is recognized that as one departs from the normal temperature of service use, criticism increases in proportion. However, in answer to criticism No. 1 it can be definitely stated that neither in the work of our own laboratory[6] or in the cooperative work of the Society's Committee D-9 on Electrical Insulating Materials has there appeared any basis to support the conclusion that there is a critical temperature at about 120 C. above which sludge formation in mineral transformer oil involves a new type of chemical reaction.

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[4] Assistant Technical Director and Chief Chemist, Research Laboratories, General Motors Corp., Detroit Mich.

[5] Chemist, General Electric Co., Pittsfield, Mass.

Items of criticism Nos. 2, 4 and 5 comprise consideration of the universality of the test procedure developed. A review of the technology of oil testing during the past decade or two will reveal that substantially no progress has been made in the development of a test for gaging sludge formation. The confusion which has resulted seems largely due to the fact that the work has ignored the fundamental chemical differences which exist between material of different sources and of different degrees and types of refining, all of which are classed under the general designation "mineral transformer oil." In studying the stability of pure chemical hydrocarbons and their derivatives, the chemist has invariably based his experimental technique on the recognized chemistry of the material under study. With mineral oil, however, the investigator has paid little if any attention to the fundamental chemical nature of the product and the variation produced by changing source and refining technique. The desire to produce a universally applicable sludge testing procedure has resulted in confusion to the point that the possibility of developing any type of sludge test is questioned. In the work of this paper, I have therefore limited the immediate objectives, believing that the accelerated oxidation and sludge formation in a mineral oil can be successfully accomplished in the light of our present knowledge only by careful recognition of the oxidation peculiarities of each type of oil. The paper is not limited, as Mr. Rosch implies, to one oil, refined in one way, from one refiner. When I say that I have limited the record of the paper to one type of oil, I mean that I have described only the behavior of the oil normally used in American transformer practice. This includes the examination of material from all the principal refiners of this country using a wide variety of refining methods. I have not attempted, however, to include, for example, all the different grades of British transformer oils including the so-called "white, non-sludging" oils. With some types of oil and some types of refining, the oxidation is sufficiently rapid to demand a reduction in the specified temperature or time factor, or both, in order to avoid the difficulties of exhaustive oxidation. It must always be borne in mind that sludge formation in mineral oil is a secondary reaction. The rapidity of the primary oxidation does not necessarily indicate the rapidity of sludge formation. If so, simple oxygen absorption tests would be the solution of the sludge testing problem. To state, therefore, that all oils have a linear rate of oxidation or that all oils can be gaged by the same sludge test is to ignore the fundamental differences existing between the numerous types of chemical compounds alone and in mixture.

With regard to a more complete demonstration of the reproducibility of the results in different laboratories and the correlation of test data with service use, I can only state that cooperative work with a limited number of oil testing laboratories shows that the method is reproducible within the close limits stated in the paper, and warrants the serious consideration of the suggested procedure by the American Society for Testing Materials, which is in an excellent position to check the reproducibility and eventually the correlation of the test with service use. It is for that purpose that the paper is presented.

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[6] F. M. Clark and E. A. Snyder, "Testing for Sludge Formation in Mineral Transformer Oil," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 568 (1936).

The chemistry of the oxidation of the various types of pure hydrocarbons gives no reason to suppose that there should exist any definite relation between the color, power factor, and sludge formation of the heterogeneous mixture called "mineral oil." The oxidation reaction is susceptible to such great changes as a result of slight changes in chemical composition and "purity" that to attempt to correlate such widely different phenomena as those responsible for sludge formation, electrical properties, and color seems unjustified.

The testing procedure has been carried out in glass vessels in order to avoid the difficulties inherent in the preparation of the surface of copper vessels. Oxidation studies have shown that to reproduce the catalytic effect of the surface of a copper receptacle is extremely difficult. Therefore glass vessels have been preferred, the catalytic effect of copper being studied by the use of a specially prepared copper wire, a new specimen being used for each new test.

SOME APPLICATIONS OF AN ACCELERATED TEST FOR DETERMINING THE CHEMICAL STABILITY OF LUBRICATING GREASES

BY F. L. WRIGHT[1] AND H. A. MILLS[1]

SYNOPSIS

An accelerated oxidation test developed to select and grade lubricating greases for chemical stability has been applied to the study of a number of conditions frequently met with in service. The test has been found of value in predicting the effects on grease stability caused by temperature changes, contact with metal surfaces or with other materials such as graphite, zinc oxide, or air-borne dirt, introduction of reinforcing agents and oxidizing inhibitors, and presence of moisture. Most of the work reported has been done on typical ball bearing greases of the soda-base type containing small amounts of lime soap. Data is presented showing that accelerated oxidation tests may be expected to provide useful information on lime and aluminum-base greases and on gear greases.

The accelerated test is not intended to take the place of service tests but may be found useful in the selection from a larger group of the more promising lubricants for such tests. It provides a means for centering attention on definitely controlled variables having to do with a particular problem. Satisfactory correlation is reported between accelerated tests and service tests in that both methods rate greases in the same order. The reliability of the accelerated test is further substantiated by the close agreement of results with well-known chemical hypotheses. To obtain good agreement it is necessary to use oxidizing temperatures which do not cause too rapid breakdown, but rather those which disclose differences within 100 to 500 hr. The selection of reinforcing and inhibiting materials to improve lubricating greases will be aided materially by the application of the accelerated test procedure to this problem.

Lubricating greases have at times been found to break down while in contact with highly finished bearing surfaces in storage or while operating in service. When greases gum, harden, become rancid and acid, or separate and bleed oil excessively, they are said to have poor chemical stability. Chemical analysis alone cannot be used to predict the occurrence of these undesirable properties in service. The cost and time involved in conducting long-time tests suggests the desirability of having an accelerated test procedure capable of selecting the most stable products from among greases designed for specific purposes, as for example the lubrication of ball bearings, and to check the uniformity of such products from batch to batch. Several accelerated test methods have been applied with interesting results to such materials as gasoline, rubber, cosmetics, plastics, fats and food products. Many of these accelerated tests make use of the rate at which oxygen is absorbed under controlled conditions, to evaluate the chemical stability of the materials under investigation.

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[1] Metallurgist, and Research Engineer, respectively, Research Laboratory, Norma-Hoffmann Bearings Corp., Stamford, Conn.

An oxygen absorption method developed in the research laboratory of the Norma-Hoffmann Bearings Corp., has been used successfully to predict the relative chemical stability of lubricating greases.[2] In principle, it measures the rate at which oxygen is used up in oxidizing the readily oxidizable constituents in the grease sample. The sample is placed in a pressure-tight steel bomb shown in Fig. 1 and the test operated under fixed conditions of temperature, pressure, surface area and weight of grease sample. Details of the procedure for conducting the test are appended.[3] Absorption data plotted to show pressure drop with respect to time in hours, indicate that the reactions taking place are usually of the autocatalytic type. An initial period of slow absorption, identified as the induction period, has been found to correspond to a similar period of stability in storage or in service. The induction period in hours is identified as the I.P. number. Some interesting applications of this accelerated test method to specific problems influencing the stability of lubricating greases are described below and absorption test data presented for a number of greases to show the possible application of the test to the study of the stability of greases other than those used in ball bearings.

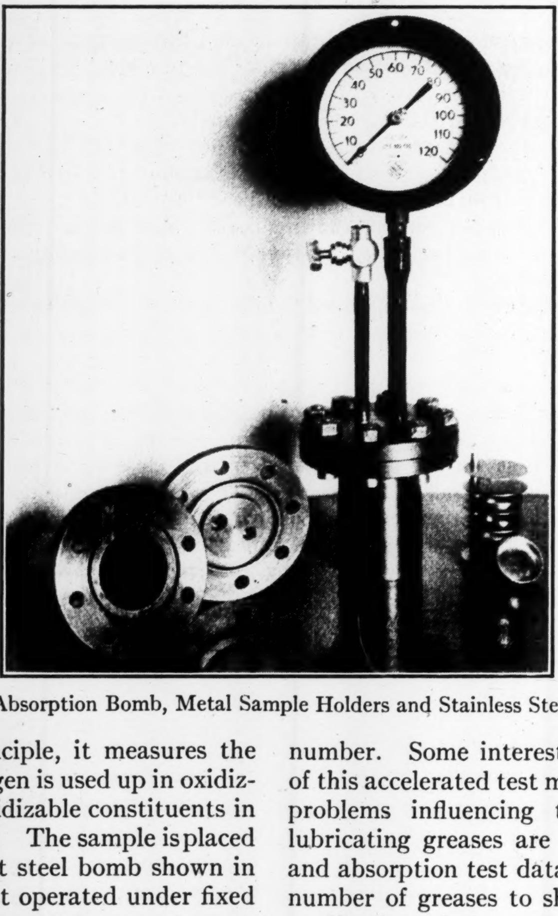

FIG. 1.—Absorption Bomb, Metal Sample Holders and Stainless Steel Rack.

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[2] F. L. Wright and W. A. Lutz, "Determining Quickly the Storage Stability of Lubricating Greases," *Product Engineering*, June, 1936, p. 210.

[3] See p. 536.

Relation of Oxygen Absorbed, to the Acid Number Developed During Accelerated Oxidation of Greases:

A typical absorption curve is shown in Fig. 2 for grease No. 1 used as a control grease (Table I) and also used in connection with the study of a number of variables discussed later. The oxidation may be interrupted at any convenient time in order to inspect the condition of the grease sample, and the absorption test may then be continued by refilling the bomb with oxygen to the pressure existing when the test was interrupted. Several check runs were made on grease No. 1 and during these runs the grease was examined at 24-hr. intervals. Other test bombs in operation at the same time supplied samples for acid-number determinations, also shown in Fig. 2 for comparison. Visual inspection of the grease showed no change in color or odor up to the end of the induction period. Shortly after the induction period had ended at 200 hr., it was observed that the grease had begun to darken and an odor indicating rancidity could be detected. At subsequent 24-hr. inspections during the period of rapid pressure drop, rancidity and darkening became more pronounced. We may conclude, therefore, that a rapid increase in acid number or a noticeable rancid odor indicates that the induction period or period of stability in storage or service has passed and that further changes in the grease may be expected to proceed at a fast rate until the readily oxidizable materials present have been exhausted.

TABLE I.—CHARACTERISTICS IDENTIFYING IN A GENERAL WAY THE GREASES DISCUSSED IN THE PAPER.

GREASE	TYPE BASE	OIL, PER CENT	SODIUM SOAP, PER CENT	CALCIUM SOAP, PER CENT	
No. 1	Soda-lime	82.0	15.0	3.0	
No. 2	Soda-lime	72.0	27.0	1.0	
No. 3	Soda-lime	88.0	11.0	1.0	
No. 4	Soda-lime	75.0	24.0	1.0	
No. 5	Soda-lime	74.0	24.0	2.0	
No. 6	Soda-lime	76.0	23.0	1.0	
No. 7	Soda-lime	77.0	21.0	1.0	1.0% oxidation inhibitor
No. 8	Soda-lime	82.0	16.0	2.0	
No. 9	Soda-lime	70.0	28.0	2.0	
No. 10	Soda-lime	73.5	24.0	2.0	0.5% oxidation inhibitor
No. 11	Lime	For use in canning machinery.			
No. 12	Lime	For use in water pump bearings.			
No. 13	Lime	For use in water pump bearings.			
No. 14	Lime	For refrigeration machinery.			
No. 15	Aluminum	Automotive shackle grease.			
No. 16	Aluminum	Automotive shackle grease.			
No. 17	Hypoid gear lubricant.				
No. 18	Hypoid gear lubricant.				
No. 19	Mild extreme-pressure gear lubricant.				

Effect of Temperature on the Length of the Induction Period:

Most chemical reactions proceed faster as the temperature increases. Consequently it is to be expected that the time required for a grease to break down will decrease with increasing temperature. That this is so, is clearly indicated in Fig. 3, showing absorption curves for grease No. 1 oxidized at 175, 210, 227.5 and 245 F. The I.P. values of several greases representing different degrees of resistance to oxidation, compared to grease No. 1, tested at these same temperatures, are shown below:

	INDUCTION PERIOD, HR.			
GREASE	OXIDIZED AT 175 F.	OXIDIZED AT 210 F.	OXIDIZED AT 227.5 F.	OXIDIZED AT 245 F.
No. 1—Control	205	48	24	12
No. 2	390	110	40	20
No. 3	650	125	64	30
No. 4	550	123	66	30
No. 5	625	150	75	33
No. 6	1200	330	150	70

These data are presented in Fig. 4 where the log of the induction period in hours is shown plotted against the oxidizing temperature. A general rule[4] for effect of temperature change on velocity of chemical reaction states that the reaction rate is about double when the temperature is increased 17 deg. Fahr. The data presented in Fig. 4 show that the temperature coefficient is of this order since the induction period is reduced by half for each 17.5 deg. Fahr. rise in temperature. Grease stability may therefore be expected to be influenced to a marked degree by small

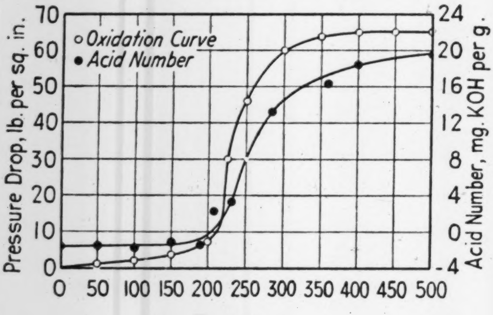

FIG. 2.—Typical Absorption Curve for Grease No. 1 and Acid Numbers Determined After Corresponding Time Intervals.

Oxidized on glass holders at 175 F., 110 lb. initial oxygen pressure.

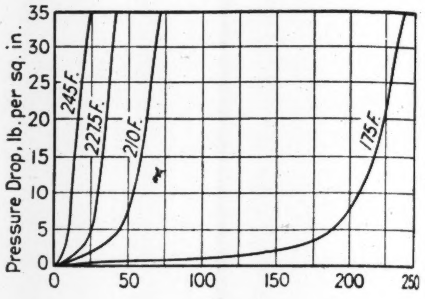

FIG. 3.—Induction Periods Shortened by Increasing Testing Temperature for Grease No. 1.

Oxidized on glass holders, 110 lb. initial oxygen pressure.

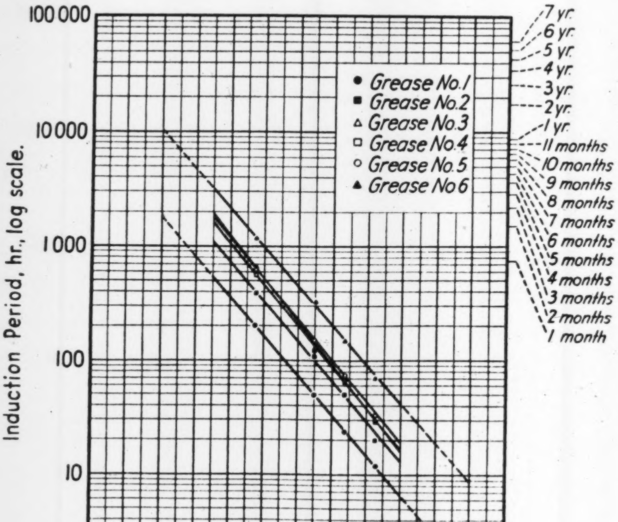

FIG. 4.—Temperature - Induction Period Curves Showing Effect of Increasing the Oxidizing Temperature on Reducing the Induction Period of Typical Ball-Bearing Greases.

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[4] Van't Hoff's Rule for Influence of Temperature on Velocity of Reaction, see "Theoretical and Physical Chemistry" by S. L. Bigelow, p. 364, The Century Co., New York City (1914).

changes in the prevailing temperature both in storage and in service. The temperature - induction period curve has been found of much assistance in extrapolating data beyond the testing range, and a single induction period determined for a grease sample at a given temperature will be sufficient to allow extrapolation over a reasonably wide temperature range. Such information obtained by extrapolation, however, is useful only for comparing one grease with another for stability under test conditions as described and is not intended to be applied directly to service conditions.

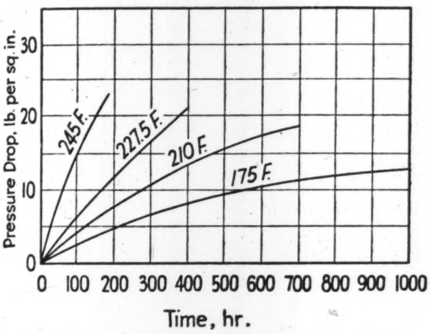

FIG. 5.—Absorption Rate Increased by Increasing Testing Temperature for Grease No. 7, an Inhibited Grease in Which the Induction Period Has Been Blocked Out.
Oxidized on glass holders, 110 lb. initial oxygen pressure.

Conclusions based on determination of the induction period apply only to commercial greases made in the usual manner from saponified animal fats or fatty acids to which mineral oil has been added. When greases are reinforced by means of powerful inhibitors or other addition agents, the induction period may sometimes be extended until it is masked or entirely blocked out. An example of this is shown in Fig. 5 for grease No. 7. Here no definite induction period developed even at 245 F., although oxidation had occurred as evidenced by an increase in acidity, discoloration and rancidity, observed at the completion of the test runs. The use of inhibitors to reduce the absorption rate and block out the induction period appears to be a promising method for improving grease stability, as discussed later. However as indicated in Fig. 5, it will be required that the rate of oxygen absorbed per unit of time be used instead of the induction period in hours as a measure of stability.

Effect on the Length of the Induction Period of Small Amounts of Metal Filings and Other Foreign Materials Introduced into the Grease:

It is well known that some metals serve to catalyze chemical reactions more than others. Since the breakdown of a lubricating grease is due to chemical action, it is reasonable to expect that metal surfaces in contact with the grease will influence the rate of oxidation. The accelerated oxidation test provides a satisfactory means for studying the effect of various metals and alloys when in contact with a lubricating grease.

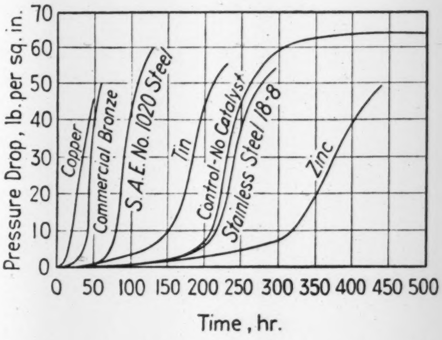

FIG. 6.—Induction Periods Varied by Introducing 2.5 per cent by Weight of Metal Filings into Grease No. 1.
Oxidized on glass holders at 175 F., 110 lb. initial oxygen pressure.

In making the oxidation test, glass dishes are used for sample holders, with no metal in contact with the grease. Tests to study the effect of metals and alloys on oxidation were made by thoroughly mixing 2.5 per cent by weight of fine metal filings into control grease

No. 1. The oxidation test was then carried out after applying the treated grease samples to glass holders in the usual manner. A few typical absorption curves for several metals and alloys are shown in Fig. 6. A list of a number of metals tested and the I.P. values for each test are given in Table II.

The copper, brass and bronze alloys are most active as is well known, but the cast iron and low-carbon steels and aluminum are also active as compared to glass. Such metals as tin, cadmium, polished hardened high-carbon steel balls, and 18 per cent chromium, 8 per cent nickel stainless steel are shown to be practically inert. The filings used were all freshly prepared and used immediately so that surface oxidation could not have occurred. In the case of purchased aluminum and zinc pellets, however, the surfaces had oxidized and it is of interest to note that such surfaces were quite inactive either in reducing the induction period in the case of aluminum or inhibiting it slightly as was the case for zinc. It is noteworthy that zinc is the only metal so far tested which definitely shows the property of retarding or mildly inhibiting the breakdown of grease No. 1.

TABLE II.—INFLUENCE OF 2.5 PER CENT BY WEIGHT OF FINE METAL FILINGS ON LENGTH OF INDUCTION PERIOD.

IDENTIFICATION OF SAMPLE	INDUCTION PERIOD, HR.
Control grease No. 1 without addition	200
Pure copper, 99.8 per cent copper	22
Phosphor bronze, 93.0 per cent copper, 7.0 per cent tin	27
Commercial bronze, 90.0 per cent copper, 10.0 per cent zinc	35
Gray cast iron	65
S.A.E. No. 1020 steel	65
Low carbon strip steel, (0.08) per cent carbon	115
Polished hard steel balls, S.A.E. No. 52,100	185
18 per cent chromium, 8 per cent nickel stainless steel	214
Aluminum strip	100
Aluminum alloy 17-S	120
Aluminum pellets (purchased, surfaces oxidized)	190
Tin, technically pure	160
Cadmium, technically pure	190
Zinc pellets (purchased, surfaces oxidized)	190
Zinc, technically pure	330

Although greases differing in stability characteristics due to variations in chemistry may not be affected in exactly the order shown, the same general trend in results has been found for other greases which have been catalyzed by these same metals. The method offers a rapid means for determining this relationship, and the simplicity of the test suggests the desirability of testing each grease for the effect of metals likely to come in contact with it in service.

Experience has dictated the use of brass and bronze alloys in the construction of many types of machine parts even though it has long been recognized that they are active catalyzers for oxidizing oils and greases. Bronze alloys are widely used in plain bearing construction to reduce friction in contact with steel and to prevent seizure by providing dissimilar metals at the rubbing surfaces. Under these conditions, it is necessary that attention be given to selecting suitable lubricating greases having the highest rating for chemical stability. The selection or development of lubricants best suited to meet the requirements imposed by a specific combination of contacting metals is ex-

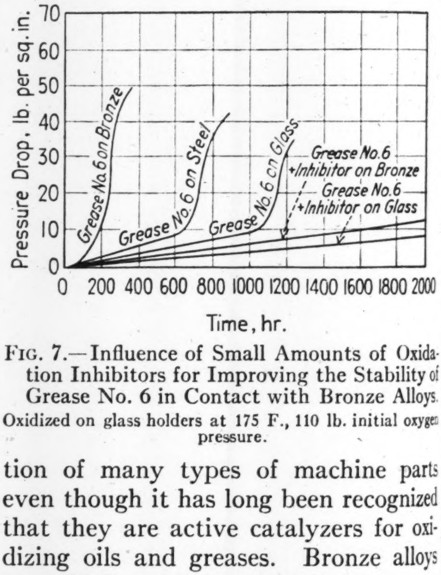

FIG. 7.—Influence of Small Amounts of Oxidation Inhibitors for Improving the Stability of Grease No. 6 in Contact with Bronze Alloys. Oxidized on glass holders at 175 F., 110 lb. initial oxygen pressure.

pedited by the accelerated oxidation test.

These severe conditions can be met only by using the most stable greases to which reinforcing agents have been added. While the use of reinforcing agents is discussed later, a specific application to inhibiting or masking the catalyzing effect of a bronze alloy is shown in Fig. 7. The absorption data for a stable grease No. 6 in contact with commercial bronze, steel, and glass are compared with similar absorption data developed after reinforcing this grease with a suitable inhibitor. After treatment, the inhibited grease is shown to be much more stable in contact with bronze than was the untreated grease in contact with steel or glass. An inspection made of the bronze metal surfaces after oxidizing grease No. 6, with and without inhibitors, showed that the untreated grease had darkened and stained the metal surface in a short time, while the inhibited grease left the bronze clean and bright after exposure for a period several times that of the untreated grease.

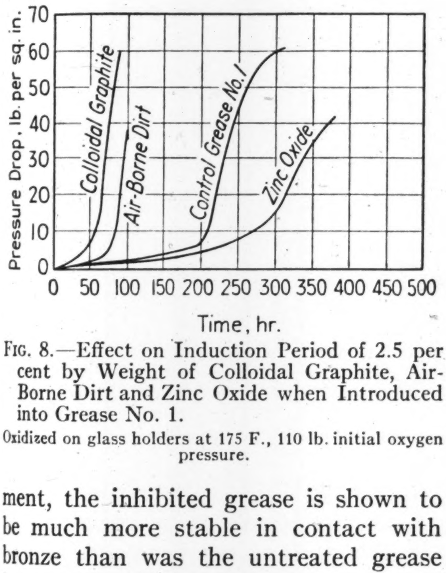

FIG. 8.—Effect on Induction Period of 2.5 per cent by Weight of Colloidal Graphite, Air-Borne Dirt and Zinc Oxide when Introduced into Grease No. 1.

Oxidized on glass holders at 175 F., 110 lb. initial oxygen pressure.

Foreign materials such as zinc oxide, colloidal graphite and air-borne dirt were investigated. Samples were prepared by thoroughly mixing 2.5 per cent by weight of these materials with grease No. 1. The results of oxidizing on glass in the usual manner are given in Fig. 8. Both dirt and colloidal graphite promote early breakdown of the grease, while zinc oxide has no harmful effect and in fact provides a definite but small inhibitive action almost identical with zinc metal. Why certain materials, including metals in fine dispersion through the grease sample, accelerate the breakdown by catalysis or direct chemical action in varying degrees while other materials and metals are passive under the same condition, is not evident from tests so far conducted.

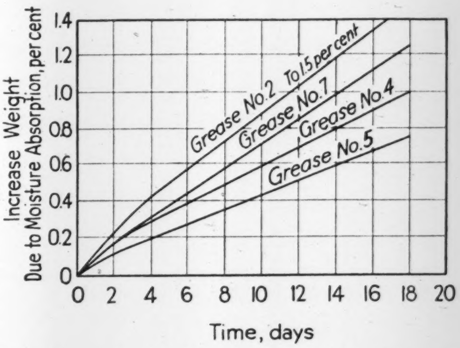

FIG. 9.—Increase in Weight Due to Moisture Absorbed by Greases Nos. 2, 4, 5 and 7 when 20-g. Samples Were Exposed to 95 per cent Humidity for 18 Days.

Effect of Moisture on Oxidation:

Preliminary tests in which water was introduced directly into the bombs in small measured amounts showed that the induction period was shortened due to the presence of moisture. A test was then made in which grease samples were weighed into glass containers in the usual manner, but before oxidizing they were placed in a humidor running at 95 per cent humidity at 70 F. for 18 days. The rate at which moisture was taken up by the grease is shown in Fig. 9 for greases

Nos. 2, 4, 5 and 7. The induction periods or absorption rates in the case of stable greases are given in Table III and show that the presence of moisture accelerates the breakdown of the grease, although the reaction is not accelerated to the extent produced by many of the active metals.

The procedure suggests a possible method for studying the rate of absorption of moisture into various types of greases and the effect of the moisture thus absorbed on chemical stability. The limited number of tests does not permit conclusions to be drawn, but the results have been introduced to indicate another application of the oxygen absorption test method. All tests involving moisture must be performed in 18-8 stainless steel bombs.

TABLE III.—THE DECREASE IN INDUCTION PERIOD OR INCREASE IN ABSORPTION RATE CAUSED BY ABSORPTION OF MOISTURE IN SPECIFIC AMOUNTS PRIOR TO OXIDATION.

	GREASE No. 2	GREASE No. 4	GREASE No. 5	GREASE No. 7
I.P. number, no moisture	460	580	480	....
With moisture	260	290	320	....
Increase in weight due to moisture, per cent	1.5	1.0	0.75	1.25
Decrease in I.P. number due to moisture, per cent	43	49	33	....
Absorption rate per 100 hr., lb., no moisture	...	...	....	1.6
With moisture	...	...	....	2.5
Increase in absorption rate due to moisture, per cent	...	...	....	60

Effect of Reinforcing Agents and Inhibitors on the Induction Period:

Although most commercial lubricating greases usually contain only the saponified fats or fatty acids and a mineral oil, much attention has been given in recent years to reinforcing lubricating greases with oiliness agents, materials to give added film strength, and inhibitors to provide greater stability. It is expected that developments leading to improved lubricating greases will take place rapidly in this field of addition agents.

Many reinforcing agents may be expected to accomplish their purpose only at the expense of chemical stability, while others may add chemical stability as well as provide the desired features, such as film strength, for which they were specifically introduced. Most important of all, addition agents, added to provide greater chemical stability, will no doubt find wide application once their value has been accepted and established. The accelerated oxidation test provides the means for determining the relative merits of the many compounds which may be suggested for this purpose, as well as predicting the amounts necessary to produce the desired optimum effects. The proprietary nature of many of the materials suggested for improving film strength and the broad patents covering many of these products, and those for stabilizing against oxidation, suggest the desirability of identifying the material to be discussed by types only. The results are introduced here to illustrate how the accelerated oxidation test method may be applied to specific problems rather than to suggest a preference for any particular reinforcing agent. Experience has indicated that each grease must be tested following addition of products suggested for use, since no general rule has developed which may safely be applied to all types of greases. The following data have been collected while carrying on a systematic survey of addition materials on typical mixed-base ball-bearing greases such as Nos. 1, 2, and 3.

Absorption tests show that unsaturated organic acids such as maleic and oleic, present in amounts as small as 0.2 per cent, reduce the induction period by one half or more. Wheat germ oils and animal oils like sperm oil containing unsaturated materials also reduce the induction period when introduced into greases in small amounts. Oxidized petroleum acids (saturated acids) are found to be inactive when introduced in amounts up to 1 per cent. Cyclic alcohols, hydroxy compounds, and organic esters have been found relatively inactive, producing very little change on the length of the induction period. Certain chlorinated compounds are definitely harmful while others are inactive. Organic phosphites and phosphates may lengthen the induction period two to three times. In a group of three proprietary products said to provide mild E.P. (extreme pressure) properties, one reduced the induction period to one quarter of its normal value, a second was almost inactive though the induction period was slightly shorter than normal, while a third product extended the induction period two to three times its original value.

The field for stabilizers of lubricating grease is almost untouched although precedent for it has been established in closely allied chemical industries such as rubber, oils and gasoline, plastics, cosmetics and certain food products. There are available a large number of chemicals which must be tried before a definite trend is established in regard to the suitability of certain types for use in lubricating greases. Some of the naphthols and amines produce a definite improvement in stability, particularly in the presence of the more active catalyzing metals, such as copper and brass. Figure 10 shows the improvement experienced when organic phosphates, naphthols and amines were incorporated into grease No. 8. It is suggested that the accelerated oxidation test be used only as a selector of the most promising products since the final choice must be based upon other practical laboratory and service tests, but even when so used, the test should become of much value in examining the effect of many hundreds of addition agents on the numerous types of lubricating greases.

Correlation of Accelerated Tests with Shelf Storage and with Performance in Service:

Two conditions are important in the grease lubrication of ball bearings, namely, stability during shelf storage and stability while bearings are operated intermittently or continuously under load. Unfortunately many changing variables are continually influencing all long-time tests and, furthermore, the end point for such tests is frequently difficult to arrive at accurately. A number of these variables have been discussed, such a changing temperature, humidity, contacting metals, and small particles of foreign materials commonly called "dirt" which may serve as nuclei for the starting point of the oxidizing reactions. Other variables such as sunlight and the film thickness of the lubricating grease in contact with the metal surfaces are known to influence long-time tests. If accelerated tests are capable of rating lubricating greases in the order of preference indicated by storage tests or service runs, then the correlation is considered satisfactory, without attempting to establish a factor for acceleration.

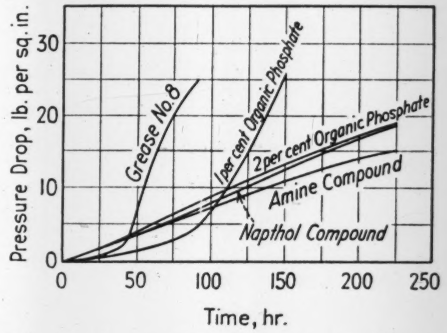

FIG. 10.—Increase in Induction Period and Decrease in Absorption Rate Due to Introduction of Mild Oxidation Inhibitors into Grease No. 8

Oxidized on brass at 175 F., 110 lb. initial oxygen pressure.

Long-time storage tests have been carried on over periods of time up to four years, and the results have in all cases substantiated the results of accelerated oxidation tests. Greases having I.P. numbers of 200 hr. or less, oxidized on glass at 175 F. and 110 lb. initial pressure, may be expected to gum, harden, or bleed oil and separate within a period of 4 to 6 months, when stored in thin films on ball bearings. Under the same conditions, greases having I.P. numbers of 1000 hr. or more have been found to remain stable for 18 to 24 months or longer. This storage condition is typical of ball-bearing-equipped electric motors which have been run in before the motors were placed in shelf storage. Grease in bearings packed in boxes or rolls and protected from air circulation and humidity by suitable packages have from 25 to 50 per cent longer shelf storage life. The prevailing temperature in storage is an important factor since it has been shown earlier that the grease life is cut in half for each 17.5 F. temperature rise, which in effect means that the life is reduced 2 to 3 times in usual summer storage as compared to winter storage at 66 to 68 F. Not all greases fail in storage due to gumming and hardening, resulting in high initial starting torque. Some lubricants fail due to bleeding of oil from soap and a thinning out in consistency as a result of oxidation so that while the starting torque is low, excessive and undesirable leaking of the oxidized oil and grease occurs. Stable greases are defined as those capable of preserving their original consistency, structure, color, and acid number after a period of months or years of storage in the machine part under examination.

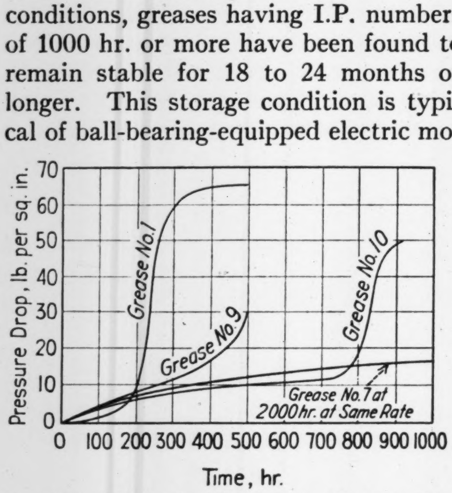

FIG. 11.—Absorption Curves for Greases Used in Service Runs at 190 F. for 2000 hr.
Oxidized on glass at 175 F., 110 lb. initial oxygen pressure.

The determination of the end point in long-time storage tests is even more difficult when service tests are performed on grease-lubricated bearings running at rated loads and speeds. Here the condition of the lubricant must be judged chiefly by visual inspection during or following the run. When high operating temperatures are involved, breakdown is accelerated, as might be expected. Correlation between accelerated oxidation tests and service runs was shown in a service test made on bearings running at 1800 r.p.m. for 2000 hr. at 190 F. Four greases having accelerated oxidation characteristics shown in Fig. 11 were rated after a 2000-hr. run at 190 F. as follows:

Grease No. 1—Badly oxidized and rancid. Thinned out in consistency and leaked from housing.

Grease No. 9—Discolored and dried out, with rancid odor, no leakage.

Grease No. 10—Slight discoloration and starting to dry out. No leakage, still usable.

Grease No. 7—Original consistency and color, with no evidence of breakdown.

Other tests carried out at normal operating temperatures of 130 to 140 F. with bearings running continuously at 8000 r.p.m. for 6 months, definitely rated the greases with short induction periods as poor and relatively unstable, while greases with long induction periods or blocked out induction periods and low absorption rates appeared best at the completion of the run.

Oxidation Data on Greases not Commonly Used in Ball and Roller Bearings:

The data so far presented have applied only to the soda or soda-lime base greases containing from 12 to 30 per cent soap, as widely used for ball and roller bearing lubrication. The control grease No. 1 is representative of a majority of commercial bearing greases. A number of greases have been observed with lower I.P. numbers and a few with induction periods several times that of grease No. 1. It is not unexpected, therefore, to find a wide difference in oxidation characteristics between greases of other types such as lime base, aluminum base and the fluid gear greases. Typical oxidation curves for several lime-base greases are shown in Fig. 12. Some have very rapid oxidation rates, some have normal I.P. numbers, and some show an inverted absorption curve peculiar to greases which have been previously oxidized, possibly in the grease-making operation itself. All these conditions are found in soda and mixed-base greases. Curves for aluminum soap greases are shown in Fig. 13, and here again the common characteristics of widely differing absorption rates are evidenced. A few of the fluid gear greases both of the mild E.P. type and the hypoid gear type were subjected to accelerated oxidation tests. These greases are characterized by low soap contents and contain reinforcing materials. Absorption curves are presented in Fig. 14 for a few of these greases used extensively in the automotive field. No information on correlation of absorption data with service has been attempted. The data have been presented to indicate that all types of lubricating greases containing soaps

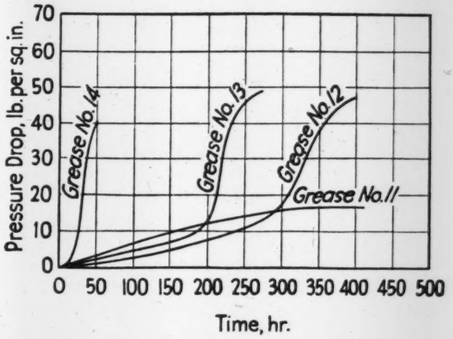

FIG. 12.—Typical Absorption Curves for Lime-Soap Greases.
Oxidized on glass at 175 F., 110 lb. initial oxygen pressure.

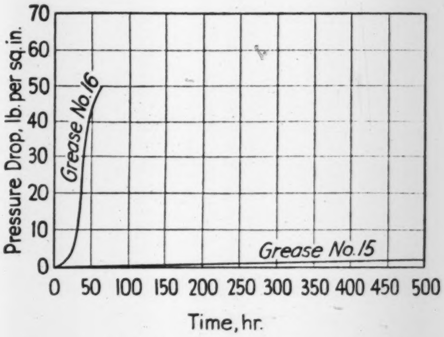

FIG. 13.—Typical Absorption Curves for Aluminum-Soap Greases.
Oxidized on glass at 175 F., 110 lb. initial oxygen pressure.

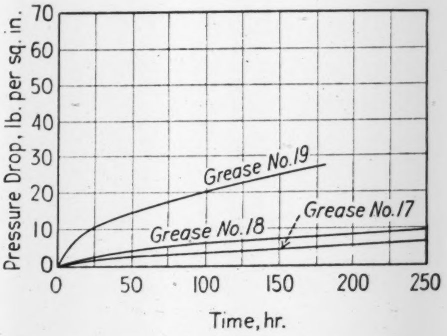

FIG. 14.—Typical Absorption Curves for Hypoid Lubricants.
Oxidized on glass at 175 F., 110 lb. initial oxygen pressure.

may be expected to react much like the ball-bearing greases.

SUMMARY

Long-time storage and service tests have demonstrated that differences exist in chemical stability between batches of lubricating greases. An oxygen absorption test method developed to disclose these differences, has been applied to the study of a number of interesting conditions encountered in service.

1. Absorption data for typical ball-bearing greases follow closely the increase in the acid number and the development of rancidity after completing an induction period, during which very little change took place.

2. The breakdown of a grease is hastened by raising the temperature. In general the induction period is reduced by half for each increment of 17.5 deg. Fahr. temperature rise.

3. A study of the effects on the stability of grease produced by its contact with metal surfaces is shown to be an important application of the test method to a specific problem. The catalyzing effect of copper-bearing alloys is clearly demonstrated and the usefulness of the accelerated test method for selecting inhibitors is shown. The catalyzing effect of gray cast iron, graphite, dirt, low-carbon steels and aluminum as compared to tin, cadmium, zinc and 18-8 stainless steel is also of much significance. The absorption method offers a means for investigating this problem with a view to explaining why these metals act as they do in the presence of various types of lubricating greases.

4. Moisture present during oxidation accelerates the breakdown of a lubricant although not to the extent produced by many metals.

5. The accelerated oxidation method provides a rapid means for investigating the effect of the numerous reinforcing and inhibiting agents suggested to improve lubricating greases.

6. Although most of the work reported has been in connection with ball-bearing greases of the soda or soda-lime base type, a few absorption tests on commonly used lime- or aluminum-base greases and gear greases show a characteristic wide dispersion in absorption rates, indicating that a study on these products would probably parallel the data presented for ball-bearing greases.

The information provided by the application of an accelerated oxidation method to the study of grease stability suggests that the method should become a useful aid in research work undertaken to develop more stable lubricating greases.

Acknowledgment:

The authors desire to express their appreciation to the management of the Norma-Hoffmann Bearings Corp. for permission to publish the results of this investigation.

APPENDIX

Test Apparatus:

The equipment consists of a number of pressure-tight steel bombs, shown in Fig. 1, made up according to details shown in Fig. 15, and a constant-temperature oil bath thermostatically controlled to provide an over-all temperature variation of not more than 1 deg. Fahr. The oil must be circulated in order to obtain the necessary close temperature control. Bombs are made of 18-8 steel and the connecting pipes are machined from beryllium copper to minimize corrosion in the presence of moist oxygen. Lead gaskets have been found best suited for use in the groove seat of the bomb chamber, to insure a good seal between the cover plate and the bomb chamber. The sample holder dishes are of glass although 18-8 steel dishes have been found equally satisfactory. The frames for holding sample dishes are made of 18–8 steel.

Test Procedure:

Sample holders and bombs must be clean and free of all contamination resulting from previous runs, or from dust settling from the air. Highest standards of cleanliness must be observed since under the accelerated test conditions traces of foreign material allowed to enter the bomb may catalyze the reaction so as to produce erratic results. The oxygen used must be of highest purity obtainable and dry. Each new tank of oxygen is checked against a standard grease to make sure no contaminating materials are present.

The grease sample is weighed to 20.00 ± 0.01 g. distributed in 4-g. samples in uniform layers in each of five sample dishes. After closing the bomb by tightening the bolts uniformly and slowly, the bomb is cleared of air by introducing oxygen *slowly* to 100-lb. pressure and releasing *slowly* four times. The oxygen pressure is raised to 100 lb. a fifth time and the bomb allowed to stand overnight to make sure there are no leaks. The bomb is then introduced into the oil bath and the pressure rise watched and oxygen gassed off until a constant pressure at 110 lb. is reached in about 2 hr. The pressure drop is observed and recorded every 2 hr., the data later being transferred to suitable curve sheets. Absorption is allowed to continue until the pressure has dropped to 55 lb., although this may be altered to suit the nature of the investigation. For convenience in tabulating a large number of results, absorption values are expressed by two numbers:

1. Induction period, I.P. in hours
2. Pressure drop, P.D. pounds drop per 100 hr. during induction period.

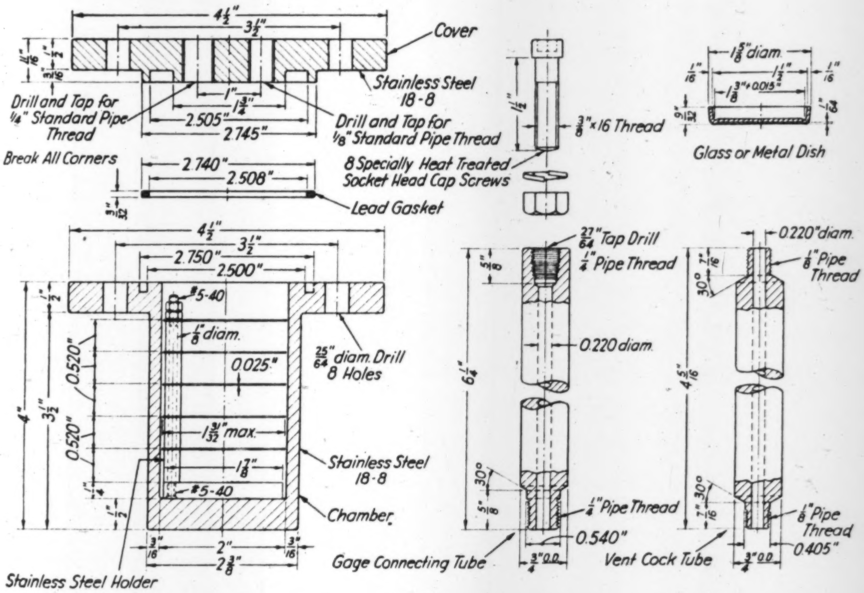
4½"
3½"
Cover
11/16"
½"
3/16"
Stainless Steel 18-8
Drill and Tap for ¼" Standard Pipe Thread
1"
1¾"
2.505"
2.745"
Drill and Tap for ⅛" Standard Pipe Thread
Break All Corners
2.740"
2.508"
3/32"
Lead Gasket
8 Specially Heat Treated Socket Head Cap Screws
1½"
⅜"×16 Thread
1⅝" diam.
1/16"
1½"
1/16"
1⅜"+0.015"
9/32"
1/64"
Glass or Metal Dish
4½"
3½"
2.750"
2.500"
½"
#5-40
⅛" diam.
0.520"
0.025"
25/64" diam. Drill 8 Holes
4"
3½"
0.520"
1 31/32" max.
1⅞"
Stainless Steel 18-8
¼"
#5-40
½"
Chamber
3/16"
2"
3/16"
2⅜"
Stainless Steel Holder
27/64" Tap Drill
¼" Pipe Thread
⅝"
0.220 diam.
6¼"
30°
⅝"
¼" Pipe Thread
0.540"
¾" O.D.
Gage Connecting Tube
0.220" diam.
⅛" Pipe Thread
7/16"
30°
4 5/16"
30°
7/16"
⅛" Pipe Thread
0.405"
¾" O.D.
Vent Cock Tube

FIG. 15.—Construction Details for Pressure-Tight Steel Bomb, Dish for Grease Sample, Connecting Gage and Vent Cock Tube, and Stainless Steel Rack for Holding Dishes During Test.

Stable greases are represented by high I.P. and low P.D. numbers for any single set of test conditions. Important information is frequently obtained by the inspection of grease samples after oxidation and for this reason a definite end point represented either by total hours of test run or after a definite pressure drop is desirable. Caution must be exercised in selecting materials which introduce no explosion hazard into the test while using oxygen at test temperatures and under pressure in the presence of oils.

DISCUSSION

MR. H. A. McCONVILLE[1] (*presented in written form*).—The method described by Messrs. Wright and Mills should be of great help to grease manufacturers in aiding them to improve their products.

Of special interest are the experiments showing the effects of small amounts of foreign materials on the stability of lubricants. The influence of these impurities is something that will have to be considered when the requirements are made more severe to produce greases which will be stable under adverse conditions. The possibilities of this method for evaluating stability of not only greases but oils and gear lubricants should be given serious consideration by all producers interested in perfecting their products and keeping abreast with the aids science can offer. It is to be hoped that Norma-Hoffmann Bearings Corp., sponsors of this work, will continue to contribute such data as will help us to make progress in the field of lubrication.

MR. J. B. RATHER.[2]—Accelerated oxidation tests in the presence of inhibitors are very frequently misleading and I do not believe that in the present state of our knowledge we can depend on accelerated tests when dealing with inhibiting agents. There seems to be a definite threshold value which determines the effectiveness of inhibitors, and when we begin to accelerate a test by raising the temperature beyond the normal operating limit, we very frequently either destroy the effect of the inhibitor or reverse the effect, so I wish to stress at this time that we should interpret accelerated oxidation tests of any type with great caution if we are dealing with inhibiting agents.

MESSRS. F. L. WRIGHT[3] AND H. A. MILLS[3] (*authors' closure by letter*).—Regarding the question concerning the correlation between long-time storage as well as service tests and accelerated oxygen absorption tests, we find a definite relationship in that the accelerated tests rate the greases in the same order of merit as do the long-time tests. No attempt has been made to establish a factor representing the extent of acceleration provided by the oxygen absorption test, chiefly because of the changing variables always met with in long-time storage or service tests.

We are in agreement with Mr. Rather concerning the care which should be exercised in interpreting accelerated oxidation tests when dealing with inhibitors and other addition agents. The temperatures selected for use in our test procedure, namely, 175 F. and 210 F. are not considered excessive since they are approached and frequently exceeded in some grease applications. We have suggested that accelerated tests may serve an important purpose in selecting the most promising inhibiting materials from among the large number of products available, but we wish to stress the point made in the paper that the final choice of a grease lubricant with or without additional products must finally depend upon the answers given by practical laboratory and service tests. We have found the oxygen absorption test to be a most useful tool in studying the stability of lubricating greases subject to the limitations of any accelerated test.

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[2] In Charge, General Laboratories, Socony-Vacuum Oil Co., Inc., New York City.

[3] Metallurgist, and Research Engineer, respectively, Research Laboratory, Norma-Hoffmann Bearings Corp., Stamford, Conn.

EFFECT OF SULFUR UPON SOME OF THE PROPERTIES OF ASPHALTS

By Isaac Bencowitz[1] and E. S. Boe[1]

Synopsis

This paper describes a method of preparing intimate mixtures of asphalt and sulfur at temperatures at which no chemical reaction resulting in the evolution of hydrogen sulfide occurs. The trend in the properties of asphalts due to the presence of sulfur is indicated. It is shown that mixtures containing 25 and 40 per cent of sulfur, when exposed to weathering age in a characteristic manner:

1. The ring-and-ball softening point does not increase as much as that of the asphalts alone.
2. The penetration of these mixtures, which is higher than that of the asphalts, drops more sharply upon aging. On remelting, the restored high penetration compares favorably with the relative restored penetration of the asphalts alone.

Mixtures made with aggregates have a much higher stability at 140 F. (60 C.) than mixtures made with asphalt without sulfur. The brittleness at 25 to 39 F. (−4 to 4 C.) is changed but little.

The use of sulfur in the manufacture of asphalts, known as the Dubbs process, has been abandoned since the advent of blown asphalts. However, various combinations of asphalts and sulfur continue to be patented. Most of these suggestions, like the Dubbs process, subject the two substances to high temperatures at which a vigorous chemical reaction takes place between the sulfur and the asphalt with the evolution of hydrogen sulfide. Asphalt technologists, on the other hand, are well aware that asphalts treated with sulfur at high temperatures weather poorly, and pavements constructed with such asphalts crack.

Preliminary work in this laboratory indicated that asphalts treated with sulfur, with strong stirring at lower temperatures and for short intervals of time, yielded products the properties of which appeared promising. It was, therefore, decided to make a careful study of this comparatively low-temperature treatment to determine in what respects the properties of asphalts are modified.

It is realized, however, that a broad problem of this nature involving asphalts is pregnant with difficulties. The methods of testing are frequently inconclusive when applied to modified asphalts for which service records are not available. On the other hand, the uses of asphalts are many, and whether it is used in the construction of pavements, roofing, or as paving brick filler, the variables involved are exceedingly complex and many are as yet not well understood.

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[1] Research Chemist, and Assistant Chemist, respectively, Texas Gulf Sulphur Co., Newgulf, Tex.

The modification of any one of the properties of asphalt does not, therefore, lead to a convincing conclusion of the utility of the change. The scope of this paper is, therefore, limited. We wish to point out that the addition of sulfur to asphalt under certain prescribed conditions modifies the original asphalts in a definite direction. We hope that these modified properties may be found advantageous for certain specific purposes.

MIXTURES WITHOUT AGGREGATE

Most asphalts will form, with 40 per cent sulfur, when heated at 300 F. (150 C.) with stirring, a mixture which will remain visibly homogeneous when cooled to room temperature. A few asphalts will form such mixtures with as much as 55 per cent sulfur. However, when the same mixtures are allowed to stand at 250 F. (120 C.) for 24 hr., some sulfur settles out. To prepare a mixture with as much as 40 per cent sulfur so that it will remain homogeneous even after 24 hr. at 250 F. requires a critical length of stirring as well as an effective stirrer.

Out of the 17 asphalts studied in this investigation, it was found that 10 will form stable mixtures (visibly homogeneous after 24 hr. standing at 250 F.) with 40 per cent sulfur after 2 hr. stirring. All of the 17 asphalts will form stable mixtures with 25 per cent sulfur. On the other hand, none of these mixtures is homogeneous in the sense of the Oliensis test.[2] When the mixtures are treated with organic solvents, most of the sulfur crystallizes out separately.

Method of Preparation.—Throughout this work all preparations were made in jacketed pots, the temperature of which was maintained at 300 F. (150 C.) by means of steam. Direct fire was not used. To make a mixture of sulfur and asphalt, a known weight of the latter is placed in the pot and as soon as it melts, the desired amount of molten sulfur is added and the two fluids are stirred for 2 hr.

All of the mixtures described in this paper were made in small batches of 200 g. The stirrer carried two blades so that one stirred at the bottom and the other a little below the upper surface of the fluids. It revolved at the rate of 325 r.p.m. However, for larger scale tests as much as 10 tons of mixtures were made in batches of 500 lb. with a stirrer revolving at the rate of 85 r.p.m.

Effect of Aging upon the Softening Point:

The effect of sulfur upon the aging properties of asphalts is obviously the most important consideration. Accordingly, mixtures were made of three different asphalts each containing 25 and 40 per cent of sulfur. Thin panels, 0.12 in. thick, of these mixtures, as well as of the original asphalts, were made on aluminum plates using the method described by Strieter,[3] and exposed to repeated cycles of accelerated weathering. The entire set of panels was prepared and exposed to the various treatments of temperature variations, rain effect and carbon arc illumination simultaneously, so that all of the panels received identical exposure.

Throughout the test, lasting about $1\frac{1}{2}$ yr., frequent examinations failed to reveal any marked distinction between the appearances of the panels other than a difference in coloring. On the other hand, the ring-and-ball softening points changed in a characteristic manner. The softening points of mixtures containing sulfur increased appreciably less than those of the asphalts without sulfur.

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[2] G. L. Oliensis, "A Further Study of the Heterogeneity of Asphalt—a Quantitative Method," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 494 (1936).

[3] O. G. Strieter, "Accelerated Tests of Asphalts," *Journal of Research*, Nat. Bureau Standards, Vol. 5, No. 1, p. 247 (1930).

Similar results were obtained when the substances were exposed in thick layers. Penetration cups were weathered in out-of-door exposure boxes similar to those described by Lewis and Hillman.[4] The data are given in Table I.

Effect of Aging upon Penetration:

The penetration[5] of mixtures containing 25 and 40 per cent of sulfur at the end of 1 hr. at room temperature after pouring and 1 hr. in the thermostat is appreciably higher than that of the original asphalt without sulfur. However, when the mixtures are exposed to out-of-door aging, the penetration of sulfur containing mixtures drops much more rapidly than is the case with asphalts alone. This apparent increase in hardness, however, is not due to permanent change in the chemistry or structure of the asphalt, for, on remelting, the original penetration of each respective mixture is almost completely restored. The results of a few characteristic mixtures are shown graphically in Fig. 1 where the penetrations are plotted against the logarithm of time in hours. The semilogarithmic scale was chosen merely to enable the representation of the data in compact form. Similar curves were obtained with all asphalts studied in this investigation.

MIXTURES CONTAINING AGGREGATES

Early in 1934 a small experimental road was built of small slabs made with sand and several asphalts with and without sulfur. The slabs were 1.5 by 3 ft. The rolling wagon was equipped with four iron wheels 2 in. wide, and was loaded with 1 ton of fire bricks evenly distributed. The wagon was pulled back and forth in the same track. It was noted that mixtures without sulfur were either too soft or crumbled, while many of those containing sulfur stood up excellently under these conditions.

TABLE I.—EFFECT OF WEATHERING UPON THE RING-AND-BALL SOFTENING POINT ASPHALTS AND ASPHALT-SULFUR MIXTURES.

Description of Asphalt[a]	Ring-and-Ball Softening Point, deg. Fahr.					
	Before Weathering			After Weathering		
	Asphalt Without Sulfur	75 per cent Asphalt 25 per cent Sulfur	60 per cent Asphalt 40 per cent Sulfur	Asphalt Without Sulfur	75 per cent Asphalt 25 per cent Sulfur	60 per cent Asphalt 40 per cent Sulfur
PANELS, 0.12 IN. THICK ON ALUMINUM PLATES EXPOSED TO ACCELERATED WEATHERING FROM DECEMBER, 1935, TO SEPTEMBER, 1937						
Texas and Mexican, Air and Steam:						
30 Penetration at 77 F. (25 C.)	168	170	174	242 243	193 221	222 224
26 Penetration at 77 F. (25 C.)	193	195	198	269 257	219 244	227
Venezuelan, Air, 28 Penetration at 77 F. (25 C.)	169	170	174	232 210	197 199	207
PENETRATION CUPS EXPOSED TO OUT-OF-DOOR WEATHERING, JANUARY, 1936, TO OCTOBER, 1937						
Texas and Mexican, Air and Steam, 30 Penetration at 77 F. (25 C.)	168	170	174	183	170	175
Venezuelan, Air, 28 Penetration at 77 F. (25 C.)	169	170	174	184	171	181
Venezuelan, Steam:						
58 Penetration at 77 F. (25 C.)	119	119	..	127	119	..
32 Penetration at 77 F. (25 C.)	133	..	134	145	..	135
Venezuelan, Vacuum, 9 Penetration at 77 F. (25 C.)	170	171	..	170	171	..

[a] The source and the method of manufacture in this column are those given by the manufacturer of the respective asphalts.

These results, though of a qualitative nature, indicated the desirability of an extensive laboratory study to establish the effect of the several variables, such as the ratio of asphalt to sulfur, the quality of sand and filler, etc. Two thousand

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[4] R. H. Lewis and W. O. B. Hillman, "A Study of Some Liquid Asphaltic Materials of the Slow Curing Type," *Public Roads*, Vol. 15, p. 92 (1934).

[5] Standard Method of Test for Penetration of Bituminous Materials (D 5 - 25), 1936 Book of A.S.T.M. Standards, Part II, p. 1080.

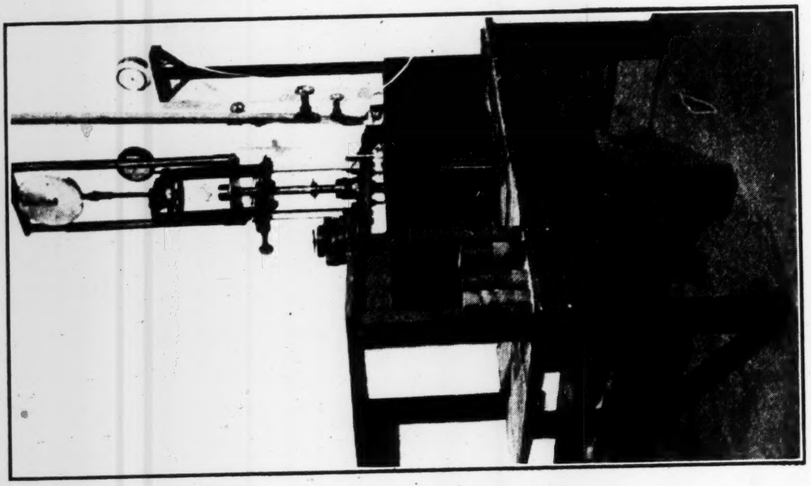

FIG. 2.—Stability Testing Machine.

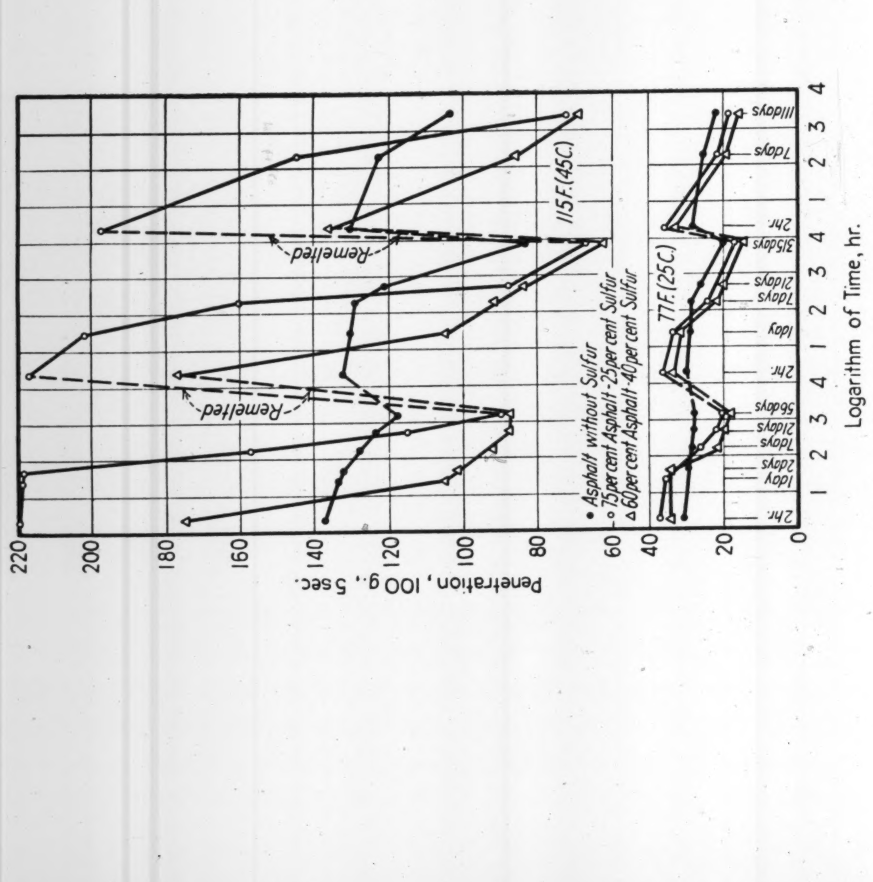

FIG. 1.—Effect of Aging upon the Penetration of Asphalt and Asphalt-Sulfur Mixtures, Air and Steam Blown. Mexican and Texas asphalt.

mixtures were studied. In this paper only a few characteristic mixtures, which represent the general trend for all the combinations investigated, are given.

Methods of Testing:

It was found desirable to develop a method of testing which would correlate the resistance to deformation at higher temperatures with the brittleness at low temperatures. The Hubbard-Field stability test was thought to be unsuitable because in some of the mixtures sulfur is "squeezed" out towards the edges, resulting in a higher stability at the periphery than at the center. On the other hand, it was thought desirable to develop a test which would give an indication as to the stability due to compression and lateral shear rather than that due to the type of shear brought into play in the Hubbard-Field stability test. The method of testing finally adopted is fully described elsewhere.[6]

For the sake of clarity a brief description of the tests employed will be included in this paper.

Stability Test.—The apparatus shown in Fig. 2 consists of a hemispherical plunger made of stainless steel of $\frac{3}{8}$-in. radius mounted so that it moves vertically under a load measured by means of a circular spring balance. The distance the plunger travels downward under the increasing load, which is equivalent to the depth of penetration into the specimen, is measured to 0.001 in. by means of a micrometer.

A briquet 2.5 in. wide, 6 in. long and 1.5 in. deep, kept in the water bath at 140 F. (60 C.) for at least 1 hr. is placed under the plunger. The plunger is brought in contact with the surface of the specimen and the load applied intermittently at the rate of 0.5 lb. every 10 sec. The depth of penetration read on the micrometer and the load read on the spring balance is recorded. The specimen is not confined by a mold, so that the material itself resists any lateral deformation.

The load required to cause the plunger to penetrate into the specimen a depth of $\frac{3}{8}$ in. is designated in this paper as the stability. However, if the load is plotted against the depth of indentation, typical stress-strain curves showing a change in the rate of deformation are obtained. The minimum rate of deformation thus shown can be used as a measure of the stability as suggested by Lee and Markwick.[7]

Brittleness Test.—Briquets similar to those used in the stability test are kept in a large refrigerator at 25 F. (−4 C.) for at least 24 hr. and then tumbled in a revolving drum for 1 hr. The percentage loss in weight after 1 hr. of tumbling is recorded as the brittleness number.

The tumbler, kept in the refrigerator at 39 F. (4 C.), consists of a cylinder 24 in. in diameter revolving at 27 r.p.m. It is perforated with a series of $\frac{3}{8}$-in. holes to allow the fines to sift out, thus avoiding abrasion due to the presence of fines. A longitudinal bar welded on the inside of the cylinder picks up the specimen and allows it to fall from the highest point at every revolution.

Flexure Test.—The equipment for testing the specimens consists of two $\frac{3}{8}$-in. roller supports set at a 5-in. span and so mounted that they could move freely both longitudinally and transversely. The load is applied by means of a small hydraulic press, and the entire assembly, including the pressure gages, has been calibrated by means of a previously tested platform scale. The deflection at the center of the specimen was indicated by means of an Ames gage.

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[6] I. Bencowitz, "Suggested Procedure in Design of Sheet Asphalt," *Industrial and Engineering Chemistry*, Vol. 29, p. 98 (1937).

[7] A. R. Lee and A. H. D. Markwick, "The Mechanical Properties of Bituminous Surfacing Materials Under Constant Stress," *Journal*, Soc. Chemical Industry, Vol. 56 p. 1467 (1937).

The bars, similar to those used in the stability test, are faced on a special grinding machine by means of a cup-shaped carborundum wheel. These are then kept at 25 F. (−4 C.) for 24 hr. and tested inside the refrigerator. The temperature of the bar during the test varied only a fraction of a degree. At the end of each test the specimen is calipered to within 0.001 in.

TABLE II.—CHARACTERISTICS OF MATERIALS USED IN THIS INVESTIGATION.

	Sand A	Sand B	Sand C	Method of Testing
Retained on No. 10 sieve, per cent by weight	0.70	......	......	
Passing No. 10 sieve, retained on No. 20 sieve, per cent by weight	5.47	3.80	3.85	
Passing No. 20 sieve, retained on No. 40 sieve, per cent by weight	55.60	17.07	17.49	
Passing No. 40 sieve, retained on No. 60 sieve, per cent by weight	35.87	22.18	22.60	
Passing No. 60 sieve, retained on No. 80 sieve, per cent by weight	1.40	22.18	22.07	
Passing No. 80 sieve, retained on No. 100 sieve, per cent by weight	0.36	17.17	16.25	
Passing No. 100 sieve, retained on No. 200 sieve, per cent by weight	0.36	16.58	16.63	
Passing No. 200 sieve, per cent by weight	0.24	0.97	1.11	
Shape of particles	Round	Round	Flat and angular	
Specific gravity	2.592	2.592	2.608	A.S.T.M. Tentative Method C 128 - 36 T[a]

ASPHALT NO. 1, TEXAS AND MEXICAN, AIR AND STEAM.

Penetration at 77 F., 100 g., 5 sec.	30	A.S.T.M. Method D 5 - 25[b]
Ring-and-ball softening point	168 F.	A.S.T.M. Method D 36 - 26[c]
Specific gravity	1.016	A.S.T.M. Method D 70 - 27[d]

[a] Tentative Method of Test for Specific Gravity and Absorption of Fine Aggregate (C 128 - 36 T), *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part I, p. 808 (1936); also 1937 Book of A.S.T.M. Tentative Standards, p. 610.

[b] Standard Method of Test for Penetration of Bituminous Materials (D 5 - 25), 1936 Book of A.S.T.M. Standards, Part II, p. 1080.

[c] Standard Method of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method) (D 36 - 26), 1936 Book of A.S.T.M. Standards, Part II, p. 1098.

[d] Standard Method of Test for Specific Gravity of Road Oils, Road Tars, Asphalt Cements and Soft Tar Pitches (D 70 - 27), 1936 Book of A.S.T.M. Standards, Part II, p. 1110.

In every other respect the procedure of testing, as well as the method of calculating the modulus of elasticity, described by Rader[8] is closely followed.

Percentage of Voids.—The percentage of voids of the compacted mixtures was determined by displacement of a 50 per cent mixture of water and alcohol.

Percentage of Water Absorption.—Briquets were kept immersed in running water for three months. The percentage gain in weight is expressed as the water absorption.

Molding the Specimen:

A given weight of asphalt is melted in a steam-jacketed pot and the required proportion of molten sulfur is added. The fluids are then stirred, and previously heated sand mixed with the proper proportion of mineral filler is added and stirred in the pot until a homogeneous mixture is obtained. The mixture is then placed in collapsible, preheated molds, tamped as described by Rader,[8] and compressed under a load of 10,000 lb. The briquets were allowed to cool for 5 min. in cold water while under pressure.

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[8] L. F. Rader, "Investigations of the Physical Properties of Asphaltic Mixtures at Low Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part II, p. 559 (1935).

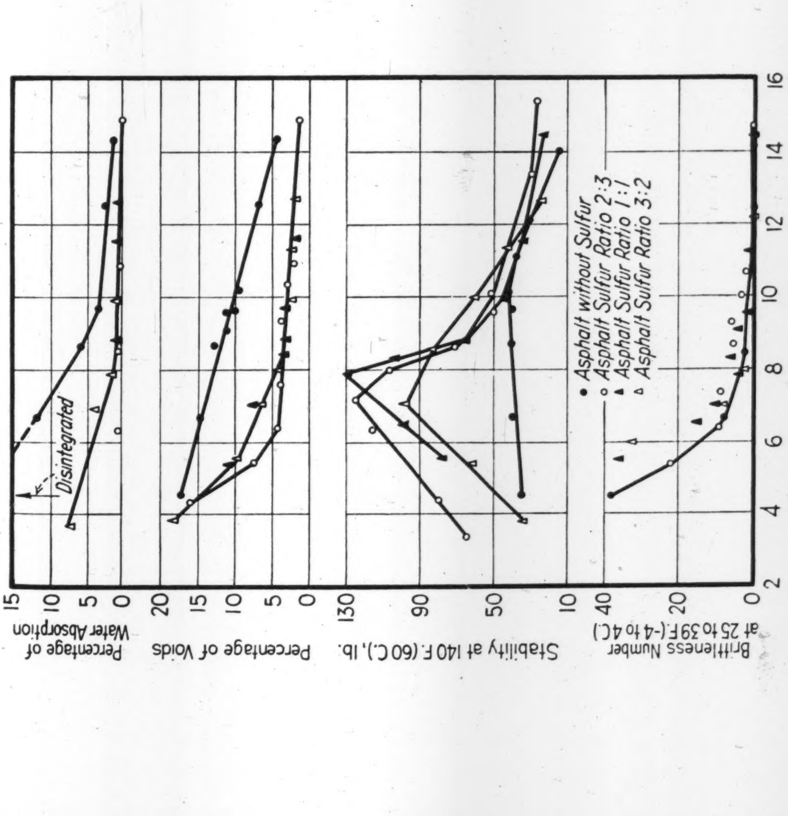

FIG. 3.—Effect of Sulfur upon the Physical Properties of Aggregate-Asphalt Mixtures. Sand A, limestone ratio, 85:15. Asphalt 1.

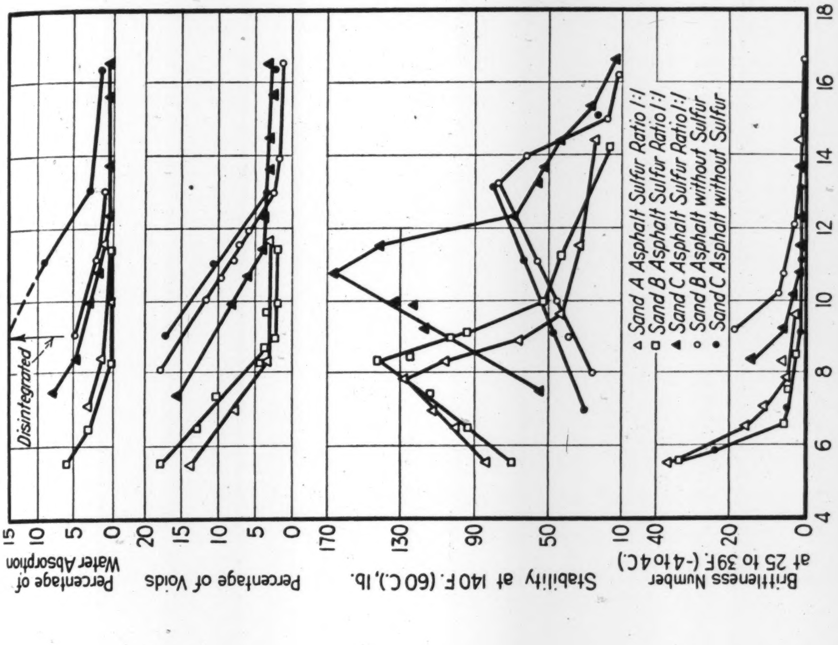

FIG. 4.—Effect of Variation of Characteristics of Sand upon Physical Properties of Asphalt Mixtures With and Without Sulfur. Sand-limestone ratio, 85:15. Asphalt 1.

Materials:

Three different sands were used in this investigation (see Table II). Sand A, a poorly graded sand, the particles of which are smooth and round, is available in this locality. Sand B was made by sieving and grading sand A. Sand C, graded similarly, was made by crushing and sieving rock. The particles of this sand were flat and angular.

Discussion of Results:

Mixtures containing sulfur cannot be compared with those without sulfur on the basis of the same weight percentage of asphalt, for obviously the weight relationship does not establish a correspondence between these mixtures. On the other hand, volume relations cannot be employed because this will involve questionable assumptions as to the role which sulfur plays in the mixture. However, since we are interested in showing only the trend of the changes due to sulfur, the entire range of compositions will be compared rather than a few specific mixtures, and, to establish the nature of the entire curve, mixtures at both extremes of compositions will be included.

In Fig. 3 is shown the effect of increasing the proportion of sulfur upon the physical properties of the mixtures made with sand A and limestone filler in the ratio of 85:15.

It will be seen that the stability at 140 F. (60 C.) due to the presence of sulfur, increases very sharply whereas the brittleness number at 25 to 39 F. (−4 to 4 C.) is not appreciably affected. Increasing the ratio of sulfur to asphalt beyond 50 per cent does not increase the stability.

In Fig. 4 are plotted the results on mixtures with different sands, the ratio of asphalt to sulfur being 1:1. It is of interest to point out that mixtures with a very poor sand A containing sulfur have a higher stability than mixtures without sulfur even when the better sands B and C are used.

TABLE III.—EFFECT OF SULFUR UPON THE MODULUS OF ELASTICITY AND MODULUS OF RUPTURE OF PAVING MIXTURES AT 25 F. (-4 C.).

Sand B, limestone filler.

Asphalt	Asphalt Sulfur Ratio	Sand Filler Ratio	Asphalt, per cent	Hubbard-Field Stability, lb.	Voids, per cent	Modulus of Elasticity, lb. per sq. in.	Modulus of Rupture, lb. per sq. in.
Mid-Continent, Steam and Vacuum, 97 Penetration at 77 F. (25 C.).........	1:0	75:25	9.0	1410	4.18	136 000	695
	1:0	75:25	8.5	1570	5.40	142 000	750
	1:0	75:25	8.0	1610	6.40	155 000	780
	1:1	85:15	8.5	1320	1.75	70 000	675
	1:1	85:15	8.0	1950	1.85	107 000	735
	1:1	85:15	7.5	2180	2.73	115 000	825
Gulf Coast and Mexican, Steam and Air, 47 Penetration at 77 F. (25 C.)...	1:0	85:15	10.6	1740	4.53	160 000	830
	1:0	85:15	10.2	2070	5.24	177 000	875
	1:0	85:15	9.5	2210	6.75	247 000	945
	1:1	85:15	8.5	2180	1.61	191 000	915
	1:1	85:15	8.0	2570	2.36	247 000	950
	1:1	85:15	7.5	3300	3.88	285 000	1005

When harder asphalts are used the trend is identical. With softer asphalts the trend is similar but less pronounced.

It is of interest to point out that mixtures with sulfur absorb appreciably less water than similar mixtures without sulfur.

Effect of Sulfur upon the Modulus of Elasticity and Modulus of Rupture at 25 F. (−4 C.):

The modulus of elasticity and modulus of rupture of paving mixtures have been correlated with service records and are considered to be measures indicating the resistance to cracking.[9] On the other hand, it is found that mixtures possessing a Hubbard-Field stability of 1500 to 2000 and a percentage of voids of 1.5 to 5.0 are satisfactory for paving purposes.[10] If we assume that this holds for mixtures containing sulfur, then we can use it as a basis of equivalence in comparing mixtures with and without sulfur.

In Table III are given data obtained with mixtures using two different asphalts, one of which is comparatively soft.

The values for modulus of elasticity are averages of three figures. The average deviation from the average is less than 5 per cent. It must be added, however, that a few briquets yielded figures which deviated as much as 25 per cent. These figures were discarded, a procedure which detracts a good deal from the precision of the data recorded. However, we feel that the trend due to sulfur is correctly represented.

SUMMARY

It is shown that the ring-and-ball softening point of mixtures containing asphalt and 25 and 40 per cent sulfur prepared at 300 F. (150 C.) does not increase as much as that of asphalts without sulfur after $1\frac{1}{2}$-yr. exposure under accelerated weathering.

The penetration of such mixtures is appreciably higher than that of the asphalt without sulfur, but on aging it decreases more rapidly. This hardening, however, is of a temporary nature since, on remelting, most of the original high penetration is restored.

A higher maximum stability at 140 F. (60 C.) can be obtained with aggregate mixtures containing sulfur without increasing the brittleness at 25 to 39 F. (−4 to 4 C.).

The maximum stability of mixtures made with a comparatively poor sand is much higher in the presence of sulfur than it is without sulfur, even when properly graded sands are used.

Acknowledgment:

The authors are deeply indebted to R. F. Bacon for his continuous interest and encouragement.

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[9] L. F. Rader, "Report on Further Research Work on Correlation of Low Temperature Tests with Resistance to Cracking of Sheet Asphalt Pavements," *Proceedings*, Assn. Asphalt Paving Technologists, January 13-14, 1937, p. 260.

[10] Prévost Hubbard and F. C. Field, "The Rational Design of Asphalt Paving Mixtures," *Research Series No. 1*, The Asphalt Inst., November 1, 1934, pp. 6-13.

DISCUSSION

Mr. H. W. Greider[1] (*presented in written form*).—The data presented by Bencowitz and Boe are extremely interesting and evidence the very great amount of careful experimental work done, but leave some questions unanswered, of which the authors no doubt are fully aware. Thus, how can one account for the fact that the large *decrease of penetration* (increase of hardness) of asphalt-sulfur mixtures on aging is not, apparently, accompanied by a correspondingly great *increase in softening point*? The asphalt-sulfur mixtures are initially softer (show more penetration) but have a higher softening point than the asphalt from which they are made—something of a paradox—but on aging they become harder (show less penetration) than the base asphalt, while the softening point increases less than that of the base asphalt. This reversal of form could hardly be predicted and is not easily explained.

The authors state that the decrease in penetration of the asphalt-sulfur mixtures is not due to permanent change in chemistry or structure of the asphalt and prove their point by substantial restoration of the original properties on remelting the weathered specimen. However, in roofing or paving asphalts such a change of characteristics would necessarily be considered permanent from the practical viewpoint, since the asphalt would not, at any time after its manufacture or installation, again be subjected to a sufficiently high temperature to cause such a restoration. Presumably, the authors have data showing the continuation of the trend indicated by the curves in Fig. 1, but where the samples were not remelted after so short a period of aging as 56 days.

It is interesting to speculate as to the physical structure of such asphalt-sulfur mixtures. Apparently, only a small part of the sulfur is in true solution in the asphalt, with the remainder dispersed as a sulfur-in-asphalt emulsion, which on cooling congeals into solid sulfur particles distributed in the plastic asphalt matrix. Since the asphalt itself is believed to consist of a complex colloidal dispersion of resins and asphaltenes in oils, the incorporation in this mixture of a liquid which can crystallize as a solid on cooling renders the system still more complicated and difficult to understand. Perhaps the crystallization of the sulfur has something to do with the changes in properties on aging noted by the authors, since this might set up a structure having high internal stress. Again, there may be a complex gel structure similar to that formed in solutions of air-blown asphalt in petroleum solvents after aging.

If one regards the preparation of asphalt-sulfur mixtures as an emulsification process, which it appears to be, the principles established for effective emulsification of other materials should be applicable. This suggests that a lower temperature of the asphalt, bringing it into the semi-fluid or plastic range during the mixing operation, would provide increased shearing stress between the asphalt and sulfur and enable thorough dispersion to be obtained in a much shorter time than two hours.

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[1] Director of Research, The Philip Carey Manufacturing Co., Lockland, Ohio.

The stability test method devised by the authors should give satisfactory data on the stress-deformation behavior of asphalt compositions, but the use of a single numerical value to express the stability may be questioned; stress-deformation curves showing the deformation rate would seem to be preferable.

The high stability shown by asphalt-sulfur paving compositions, particularly at 140 F., is very interesting. High stability can also be obtained by incorporating finely-divided, inert mineral fillers in the asphalt and such fillers would have the advantage that, due to their chemical and physical inertness and lack of tendency to crystallization or other fundamental structural change on aging, the properties of the composition would remain more nearly those of the mixture originally made.

MR. ISAAC BENCOWITZ.[2]—Mr. Greider raised two questions which we have tried to answer experimentally. Unfortunately we must admit that our answer is as yet not very convincing.

We feel that the question of aging is simplified if we differentiate between permanent and temporary aging. Penetration is a measure of both, whereas a change in the ring-and-ball softening point, since the procedure involves remelting, is a measure of permanent hardening. This itself would have been academic, and as Mr. Greider points out, of little interest to the roofing and paving technologist had it not been for the fact that permanent aging is indicative of deterioration, or breaking down of the asphaltic structure, whereas temporary hardening is some function of thixotropic properties. This rapid, temporary hardening of asphalt-sulfur mixtures may be decidedly advantageous in various fields of application.

The second question as to the function of sulfur in the asphalt-sulfur mixtures is more difficult to answer. In the accompanying Fig. 1 we have plotted the penetration against the percentage of various fillers added to asphalt. It will be seen that sulfur mixtures behave in a characteristic manner, quite distinct from mixtures containing such fillers as silica dust or carbon black.

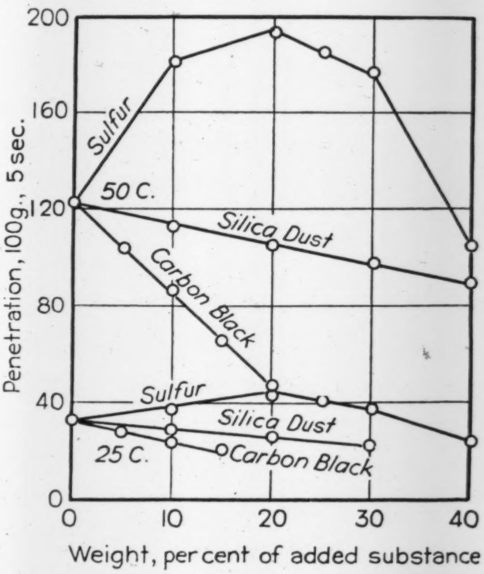

FIG. 1.—Variation of Penetration of Asphalt with the Proportion of Added Substance. Air and steam blown, Mexican and Texas asphalt of penetration 30 at 77 F.

It may be of interest to add that whatever the function of sulfur in these mixtures may be, it has been established with some degree of certainty that the presence of sulfur endows the mixtures with new properties. That these properties may be useful is shown by the fact that one of these mixtures was found to be a promising paving brick filler in a test conducted by the Ohio Department of Highways in 1935[3] (Litehiser, R. R., and Schofield, H. Z.,

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[2] Research Chemist, Texas Gulf Sulphur Co., Newgulf, Tex.

"Progress Report On Brick Road Experiments in Ohio." *Proceedings* of the Sixteenth Annual Meeting of the Highway Research Board, November, 1936, Page 182). At the end of one year's service the specification of this material was adopted by the Ohio Department of Highways.

Sulfur-asphalt mixtures have shown a good deal of promise in other fields of application which we hope to describe in a future publication.

A MACHINE FOR TESTING HIGHWAY SUBGRADE SOILS

By E. O. Rhodes[1]

Synopsis

In order that stabilization of highway subgrade soils may emerge completely from the experimental stage, better construction devices and practices are needed in the field and better methods of test are needed in the laboratory.

In general, the laboratory testing of highway subgrade soils involves determinations of (1) mechanical analyses; (2) soil constants; (3) optimum moisture content for maximum density; and (4) quantity of stabilizer required as indicated by shear or alternate wetting-and-drying or freezing-and-thawing tests.

These determinations, while helpful, are not entirely satisfactory, because (1) mechanical analyses are time consuming and do not necessarily correctly indicate suitability for highway subgrade purposes; (2) soil constants are determined by empirical methods having poor reproducibilities and doubtful significances; (3) optimum moisture determinations vary with the method and amount of compaction employed and can only approximate service results using field equipment; (4) shear, wetting-and-drying, and freezing-and-thawing tests usually are performed with static, compacted samples after immersion in or contact with water. There is no satisfactory agreement between the results of these laboratory tests and service behaviors apparently for the reason that the tests do not include the element of motion which is imparted by traffic on the road.

A mechanical device, called the Plastograph, has been located and tested which, apparently, correctly indicates the effect (service behavior) of continued manipulation (traffic) on different soils (roads) in the presence of varying amounts of water (rain). It consists of a miniature pug-mill, driven by a synchronous motor, through a dynamometer arrangement which measures and records the resistance of the material under test to mechanical force (viscosity in gram-meters) as a function of time.

Tests to date indicate that the use of the Plastograph in soil studies will make it possible to predict with greater certainty the behavior of soils to be used for highway purposes with or without admixtures of stabilizing agents.

The stabilization of highway subgrade soils with admixtures of coal tar and other soil stabilizers has made notable progress. However, much remains to be accomplished, both in the field and in the laboratory, before it can be said that this new and important branch of highway engineering has emerged completely from the experimental stage.

In the field, better construction practices and improved mechanical devices are needed, and in the laboratory new and better methods of test must be developed or the ability properly to interpret present test results must be realized. For example, the author is convinced that further progress in the use of coal tar as a stabilizing agent will be greatly accelerated if and when highway and commercial laboratories are provided with methods of test and test equipment which will more accurately evaluate soils, both before and after adding coal tar, than do present laboratory methods.

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[1] Technical Director, Tar and Chemical Division, Koppers Co., Pittsburgh, Pa.

In general, these methods may be divided into four groups as follows:

1. Mechanical analysis by sieve and hydrometer methods.
2. Determination of soil constants: plastic limit, liquid limit, shrinkage limit, field moisture equivalent and centrifuge moisture equivalent.
3. Determination of optimum moisture content for compaction to maximum density.
4. Determination of the quantity of tar required for stabilization by means of shear and accelerated weathering tests.

In the laboratories of the Koppers Company, Tar and Chemical Division, the following procedure is followed when a soil of unknown service behavior is evaluated as a highway subgrade material:

A representative sample of the soil under consideration is prepared for test in accordance with A.S.T.M. tentative method of preparing soil samples for analysis (D 421 - 35 T).[2]

The prepared sample is then tested in accordance with the following A.S.T.M. tentative methods:[3]

Mechanical analysis (D 422 - 35 T)
Liquid limit (D 423 - 35 T)
Plastic limit and plasticity index (D 424 - 35 T)
Field moisture equivalent (D 426 - 35 T)
Shrinkage factors (D 427 - 35 T)

The optimum moisture content for compaction to maximum density is determined in the following manner:

Sixteen-gram portions of the dry soil (passing No. 10 sieve) are mixed with varying percentages of water, each mixture containing, in addition to the water, 4 per cent by weight of tar (based on dry soil). Since tar aids in compacting, an average amount is used when performing this test. From each mixture two briquets, each containing 8 g. of dry soil, are formed by compaction in 1-in. diameter molds under a static load of 300 lb. (This procedure was selected to approximate field conditions using a sheepsfoot roller for compacting.)

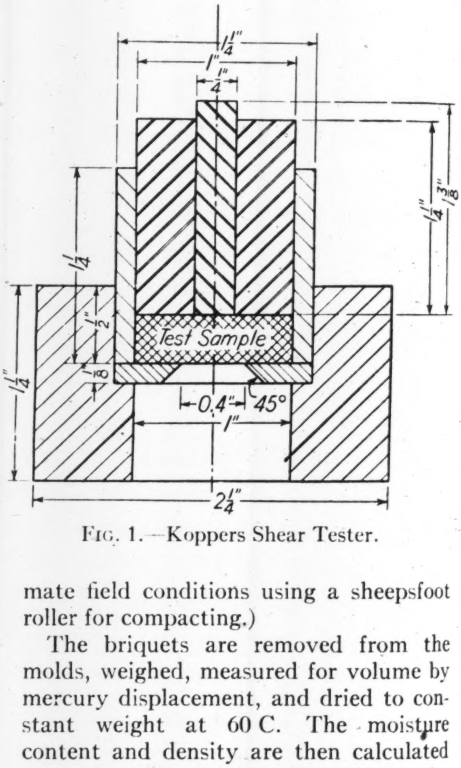

FIG. 1.—Koppers Shear Tester.

The briquets are removed from the molds, weighed, measured for volume by mercury displacement, and dried to constant weight at 60 C. The moisture content and density are then calculated using the following formulas, the results being averaged for each pair of briquets:

$$\text{Moisture (per cent by weight)} = \frac{\text{Wet wt.} - \text{Dry wt.}}{\text{Dry wt.}} \times 100$$

$$\text{Density (lb. per cu. ft.)} = \frac{\text{Dry wt. (g.)}}{\text{Wet volume (cu. cm.)}} \times 62.4$$

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[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 950 (1935); 1937 Book of A.S.T.M. Tentative Standards, p. 882.

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, pp. 953, 966, 970, 976, 978, respectively (1935); 1937 Book of A.S.T.M. Tentative Standards, pp. 885, 898, 902, 908, 910, respectively.

Plotting moisture contents (abscissas) against corresponding densities (ordinates) produces a curve, the high point of which indicates that moisture content at which maximum density may be obtained with the given compaction procedure.

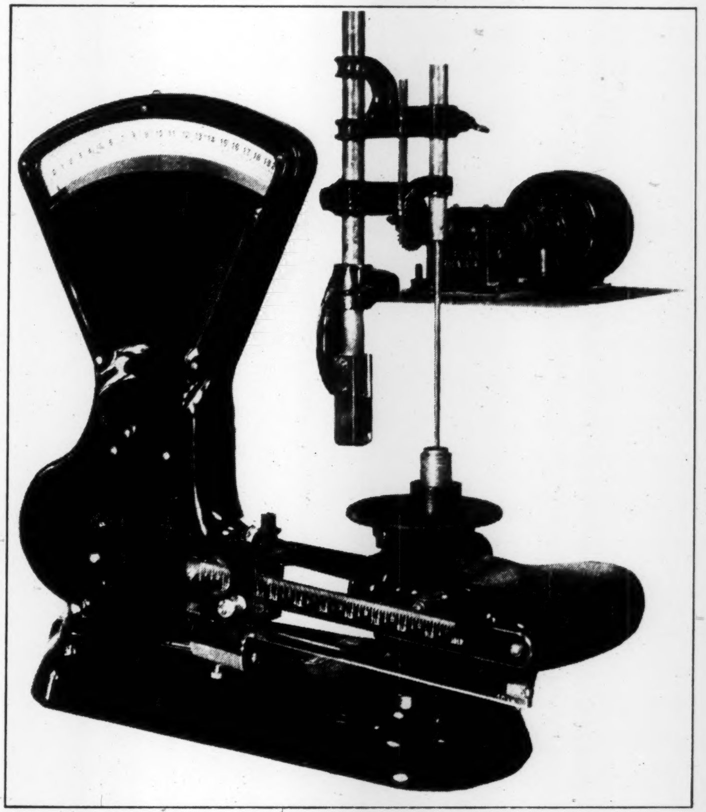

FIG. 2.—Testing Unit on Weighing Scale.

The above tests determine the optimum moisture content for -10 mesh material. This is adjusted to a total soil basis in the following manner:

Percentage of moisture in total soil for compaction to maximum density = percentage of moisture for maximum density for -10 mesh soil × percentage passing No. 10 sieve + 3 per cent × percentage retained on No. 10 sieve.

The amount of tar required for satisfactory stabilization of the soil is determined on the fraction passing a No. 10 sieve. A series of soil-water-tar mixtures, each containing 32 g. of dry soil, is made by mixing the soil at optimum moisture content for maximum density with varying percentages of tar. From each of the mixtures four specimens are made by compacting in 1-in. diameter molds, under a static load of 300 lb., a sufficient amount of each mixture so that each specimen contains 8 g. of dry soil. Specimens are then removed from the molds, weighed, and oven-dried to constant weight at 60 C. Specimens are then immersed in water 18 hr., removed, surface dried with absorbent paper, and tested for stability in a Koppers Shear Tester No. II (Modified Hubbard Field Type). The test is performed in the following manner:

The specimen is inserted in the shear tester and the assembled testing unit (Fig. 1) is placed on a weighing scale (Fig. 2), calibrated in ounces. Force is applied to the test specimen by means of a constant speed motor and gear arrangement fastened directly over the scale. The gear is connected to a piston which moves downward at a rate of 3 in. per min. and exerts a constantly increasing force through a ¼-in. diameter plunger on the test sample. The magnitude of the force is given by the indicator on the scale. When the force reaches a certain magnitude which is governed by the character of the stabilized specimen, a shearing action takes place and the center core is forced through an orifice having a diameter of 0.4 in. When this breakdown in the structure of the specimen takes place the indicator on the scale shows a sudden decrease in force; the maximum reading on the scale is taken as the stability of the test specimen.

The results obtained from each group of four specimens are averaged to obtain the "stability" for each of the various mixtures. A curve is obtained by plotting percentage of tar against stability in ounces. A stability value of 225 oz. on the curve determines the percentage of tar required for stabilizing the fraction of soil passing a No. 10 sieve. This percentage is then adjusted for the total soil sample in the following manner:

Percentage of tar required for total soil sample = percentage of tar determined from test × percentage of soil passing No. 10 sieve.

The percentage of tar required to stabilize the soil is then converted into gallons of tar per square yard per inch of compacted depth, as follows:

Gallons tar per square yard per inch of compacted depth = $0.077 \times A \times B$.

where

A = Percentage of tar required for total soil sample, and

B = Density in pounds per cubic foot.

$$0.077 = \frac{27\text{ cu. ft. per cu. yd.}}{36\text{ in. per yd.}} \times \frac{1}{9.7\text{ lb. tar per gal.}}$$

Total gallons tar per square yard = gallons per square yard per inch of compacted depth × compacted depth in inches.

Use of the above test procedure in connection with our soil stabilization investigations has been helpful but not entirely satisfactory for the following reasons:

Determinations of mechanical analysis are reproducible with a fair degree of accuracy but they are time-consuming and do not always indicate correctly whether certain soils are better or worse than others for highway subgrade purposes. It is possible for a given soil to have a mechanical analysis within the preferred limits for good soil mortars and yet be unsatisfactory as a highway subgrade material.

The methods used for the determination of soil constants are entirely empirical and, especially when performed by different operators, are not highly reproducible. They, together with mechanical analyses, are used for classifying soils roughly into different types and to that extent indicate in a general way probable field behavior and possible need for stabilization. However, due to their poor reproducibilities and doubtful significances, they do not furnish data that are sufficiently accurate to permit accurate comparisons to be made. Frequently we have found that soils having almost identical mechanical analyses and soil constants require different amounts of tar to meet the Koppers shear test described above, indicating that one or more of these tests are inaccurate or that there are some chemical or physical properties which affect the stabilization with tar that are not indicated by mechanical analyses or soil constants.

Any determination of optimum moisture for compaction to maximum density which depends upon a compaction method is affected by the method and amount of compaction employed. On this account, laboratory determinations can only approximate service results using field equipment. Our test, described above, is subject to this criticism. Also like others of similar nature, our test may be criticised because it is performed on a portion of the total soil and adjustment to a total soil basis must be made by calculation. It has some advantages over other methods in that it employs small samples, can be performed more quickly and with a higher degree of reproducibility.

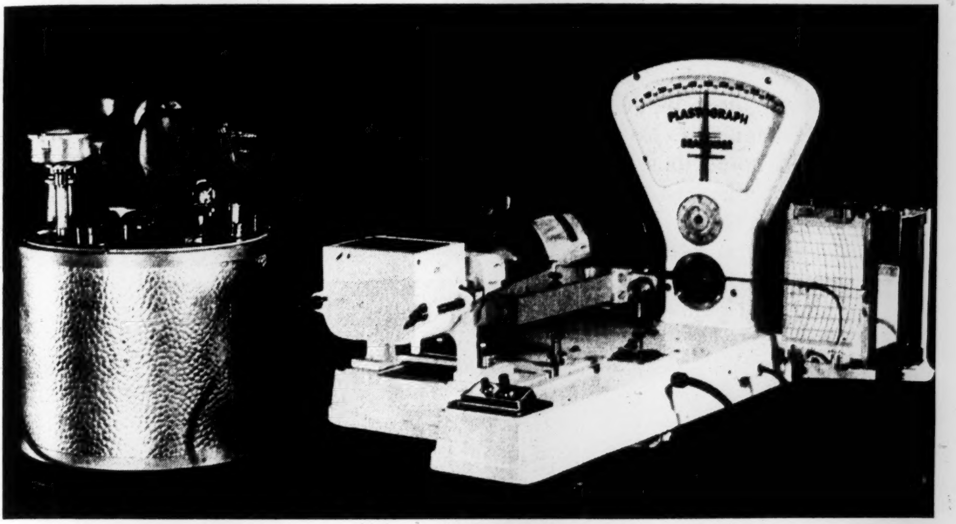

FIG. 3.—Plastograph.

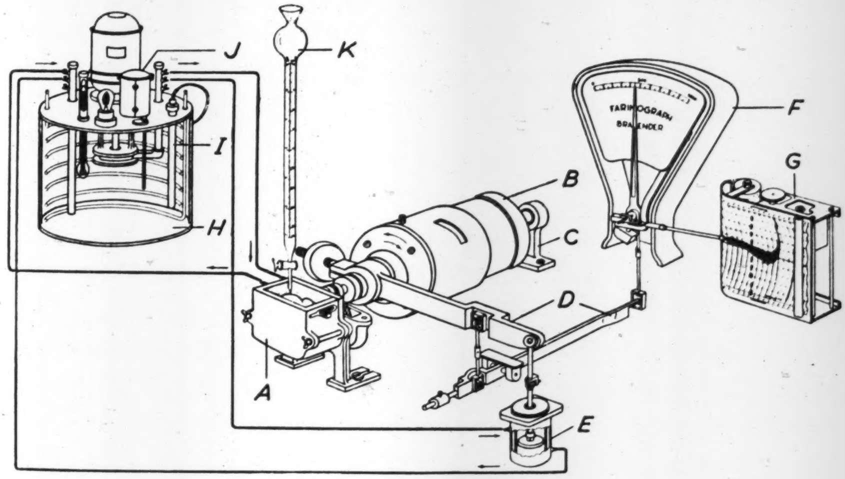

FIG. 4.—Diagram of Plastograph.

Shear tests, in general, when applied to stabilized soils are intended to measure their shear resistances after immersion in or contact with water for given periods of time. In the Koppers shear test, as in other tests of similar nature, the samples are not disturbed during the period of water absorption and are, therefore, unlike compacted soils in highway subgrades which are manipulated by traffic, while water is being absorbed. We believe this difference accounts for the fact that some clay soils which have high stability values, as indicated by laboratory shear tests after immersion in water, fail when subjected to field conditions.

Alternate wetting and drying and alternate freezing-and-thawing tests are sometimes employed in addition to or in place of shear tests to more nearly simulate field conditions, but, in addition to being time-consuming and difficult to interpret, they are static tests and lack the manipulation to which highway subgrade soils are subjected.

Recently we came to the conclusion that some test is needed that will measure or indicate the behavior of soils when manipulated in the presence of water. We had in mind the possibility of performing in the laboratory some test which would simulate the action of traffic on a road surface during the continued application of water by rain. We know that certain topsoils and sand-clay mixtures, when used on the surfaces of roads, will remain passable under moderately heavy traffic, even when subjected to heavy and continued rainfall. On the other hand, we know that many clay roads will deform badly or become entirely impassable under traffic if they are subjected to even moderately heavy rainfall, but if they are not subjected to traffic while they are wet they are still satisfactory after the water has evaporated. It seemed to us that if a machine could be located or developed which would indicate the effect (service behavior) of continued manipulation (traffic) on different soils (roads) in the presence of varying amounts of water (rain), significant and measurable differences might be determined for soils known to have good and bad service behaviors. With such a device it should then be possible to evaluate highway subgrade soils more accurately and to study and measure the effects of different stabilizing agents.

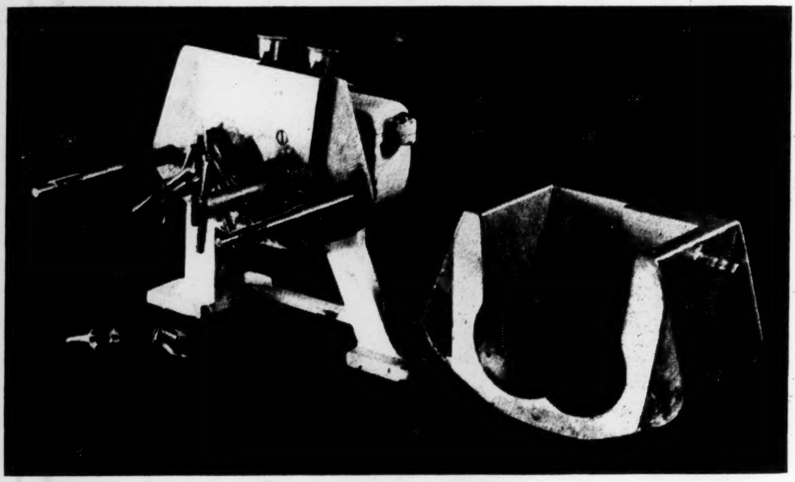

FIG. 5.—Pug-Mill Type Mixer.

We now believe that we have located such a machine. At present it is manufactured only in Duisburg, Germany, by the Brabender Corp., but within a few months it will be produced in the United

States as well. In the flour milling and baking industries for which it was developed, it is called a Farinograph. When used for other purposes it is called a Plastograph. The latter name will be used hereafter in this presentation.

The appearance of a complete plastograph unit is shown by Fig. 3. Its operation can best be explained by referring to Fig. 4. A small mixer *A* is driven by a synchronous motor *B* which is so mounted *C* that it is free to rotate. Each resistance which the mixing blades encounter in the material under test asserts itself as a reaction force in the motor casing and tends to rotate the latter in the opposite direction. This turning movement in the motor casing is transmitted to a weighing system *F* and to a recorder *G* by means of a lever system *D*, whose oscillations are restricted by a dash-pot *E*. The resistance of the material under test to mechanical force is recorded on a chart as a function of time. By varying the temperature of mixing, by means of a circulating thermostat *H*, resistances to mechanical force at different temperatures may be measured.

Believing that certain soils may behave somewhat like flour in the presence of increasing amounts of water, arrangements were made to test a few soil samples at the Brabender Corp. laboratories in New York. The results were not entirely satisfactory but they were so encouraging that additional samples were sent to the factory in Duisburg, Germany, and were tested there in the presence of the author.

For the tests in New York a standard Farinograph mixer was used. Its mixing blades were of the dough mixer type. For the tests in Germany a mixer of the pug-mill type (Fig. 5) was employed. The results with this mixer were so satisfactory that a complete plastograph equipped with a similar mixer was purchased for use in America. It was received in April and since then has been in constant service in our Pittsburgh laboratories.

Three different soils were tested in Germany:

A—A topsoil of good quality furnished by Mr. W. H. Mills, Jr. of the South Carolina Highway Department (8-213).

B—A moderately unstable clay soil from a tar stabilization project between Johnsonville and Hemingway, South Carolina (8-537).

TABLE I.—MECHANICAL ANALYSES AND PHYSICAL CONSTANTS FOR THE THREE SOILS TESTED.

	Soil A (8-213) South Carolina Top Soil	Soil B (8-537) South Carolina Clay Soil	Soil C (8-536) Georgia Clay Soil
Sieve analysis:			
Percentage passing No. 10 sieve	100	100	100
" " " 20 "	64	98	98
" " " 40 "	44	93	94
" " " 60 "	35	86	86
" " " 140 "	27	56	72
" " " 200 "	24	53	68
Liquid limit	12	27	44
Plastic limit	0	16	28
Field moisture equivalent	12	26	37
Shrinkage limit	14	14	22

C—A very unstable clay soil from a tar stabilization project in Pine Mountain Valley, Georgia (8-536).

Also, mixtures of soil *B* with 2 and 4 per cent tar and mixtures of soil *C* with 2 and 4 per cent tar were tested in Germany.

The mechanical analyses and physical constants for soils *A*, *B*, and *C* are given in Table I.

They were tested by the plastograph in the following manner: A 300-g. portion of the dry soil was placed in the mixer, the chart adjusted to a zero position and the motor started. Water was then added uniformly and continuously at the rate of 3 ml. per min. When soil

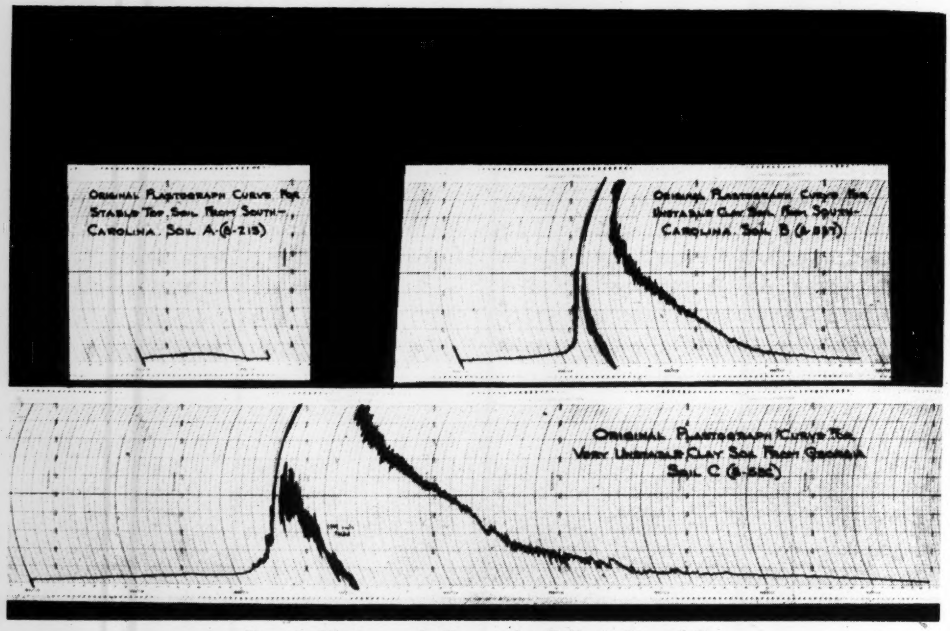

FIG. 6. Plastograph Curves for Soils A, B and C.

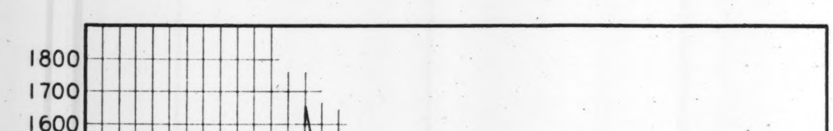

FIG. 7.—Plastograph and Size Gradation Curves for A Stable Top Soil (8-213), B Moderately Unstable Clay Soil (8-537), and C Very Unstable Clay Soil (8-536).

adhered to the sides of the mixer above the mixing blades it was dislodged and returned to the soil-water mixture. A curve representing consistency in gram meters *versus* time in minutes was plotted on the chart by the recording device. Since the rate at which the water was added was 3 ml. or 3 g. per min. and the original weight of the dry soil sample was 300 g., the abscissas of the curves may be read either as minutes or as water per cent by weight of the original dry soil.

The original plastograph curves for soils *A*, *B*, and *C* are shown in Fig. 6. To facilitate comparison and to show correctly the slopes of the curves which are distorted by the curved ordinates of the plastograph charts, the original curves were replotted with rectangular coordinates, as shown in Fig. 7. Cumulative grain size curves, plotted on a soil gradation chart, are also included in Fig. 7 in order that they may be compared readily with the replotted plastograph curves.

The plastograph curves indicate that soil *A* is not appreciably affected by water, that soil *B* is affected to a greater extent and that soil *C* is affected more than either of the others. This is in agreement with their known service behaviors.

In these three cases, the positions of the cumulative grain size curves, with respect to the preferred grading area for good soil mortars, are also in the same order as their respective behaviors when wet, as indicated by their service records and also by their plastograph curves. From this it might be inferred that cumulative soil gradation curves alone may correctly indicate the relative behaviors of different soils in the presence of water. However, experience has shown that this is not always true. Some soils whose gradation curves lie within the preferred grading area for good soil mortars behave unsatisfactorily when wet. This is also shown by some of the plastograph curves which are discussed in later portions of this presentation.

Figure 8 shows how the plastograph makes it possible to determine whether an unstable soil can be made more stable by the addition of coal tar. The curves in Fig. 8 were replotted from plastograph curves obtained in Germany, using samples prepared from soil *B* in America, in the following manner: Portions of soil *B* were mixed with sufficient water to make them easily workable. Then tar was added in the proportions of 2 per cent and 4 per cent by weight on the basis of dry soil, and the soil, water, and tar were mixed thoroughly, dried and pulverized. Three hundred-gram portions of these pulverized mixtures were tested in the plastograph in the manner described above. The replotted plasto-

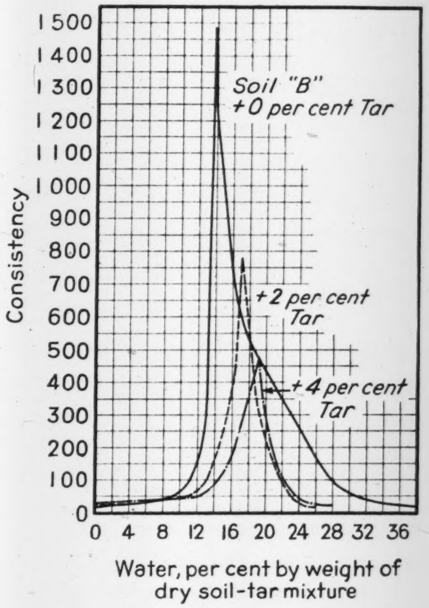

FIG. 8.—Plastograph Curves for Moderately Unstable Soil B (8-537) and Mixtures of Soil B with 2 and 4 per cent Coal Tar.

graph curves show that an unstable soil can be made more stable by the addition of coal tar. They also indicate that the amount of stabilization varies with the quantity of tar employed as a stabilizing agent.

Another case of the same kind is shown by Fig. 9. Soil *C* came from a road in Pine Mountain Valley, Georgia, which was very unstable in wet weather before the addition of coal tar in 1936. Comparison of the curves in Fig. 9 with those in Fig. 8 shows that a given quantity of tar was less effective when added to very unstable soil *C* than it was when added to moderately unstable soil *B*. This same difference was noted in the field.

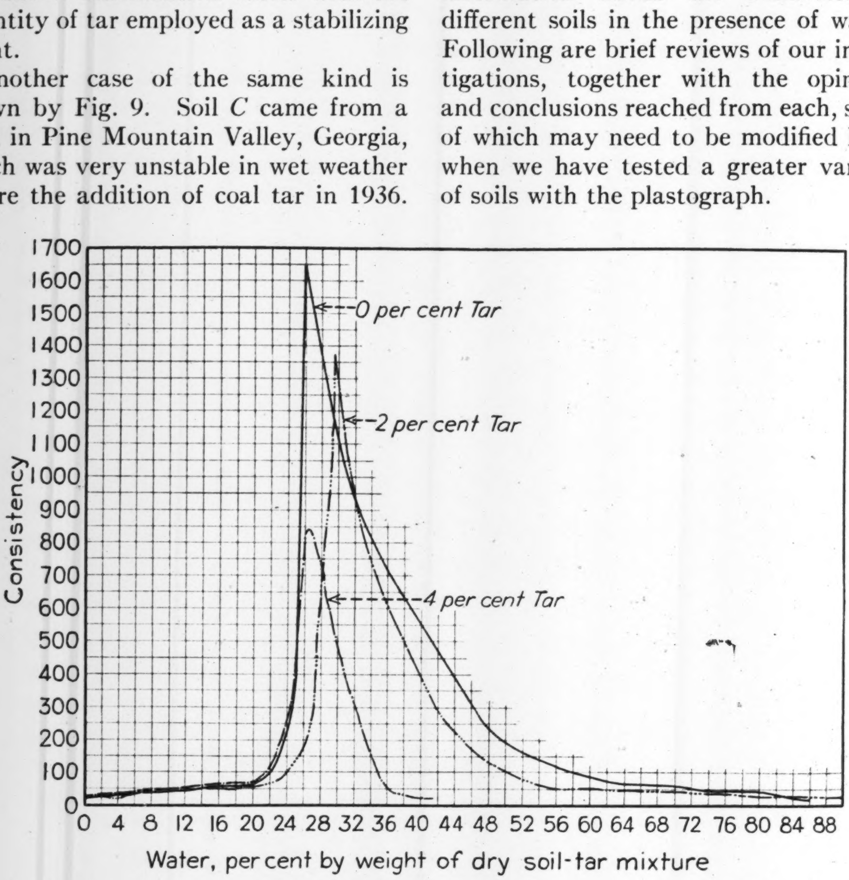

FIG. 9.—Plastograph Curves for Very Unstable Soil C (8-536) and Mixtures of Soil C with 2 and 4 per cent Coal Tar.

The plastograph tests discussed above were all conducted in Germany. When our plastograph was received in Pittsburgh we started a series of investigations which was intended to determine to what extent a plastograph can be used in soil studies. At the same time we expected to obtain some fundamental information about the behaviors of different soils in the presence of water. Following are brief reviews of our investigations, together with the opinions and conclusions reached from each, some of which may need to be modified later when we have tested a greater variety of soils with the plastograph.

Investigation No. 1—Fig. 10:

In order to determine which portions of a −10 mesh soil are affected most by the addition of water, we separated soil *A* (good topsoil from South Carolina) into the following sieve fractions: 10 to 20, 20 to 40, 40 to 60, 60 to 140; −140, and −200 mesh. Three hundred gram portions of each fraction and of the original −10 mesh topsoil were then tested with the plastograph in the standard manner and the results were replotted as shown in Fig. 10.

The 10 to 20 mesh fraction had a high consistency at the start, due probably to friction between the coarse sand particles, but water served as a lubricant and its consistency decreased in a regular manner with the addition of water.

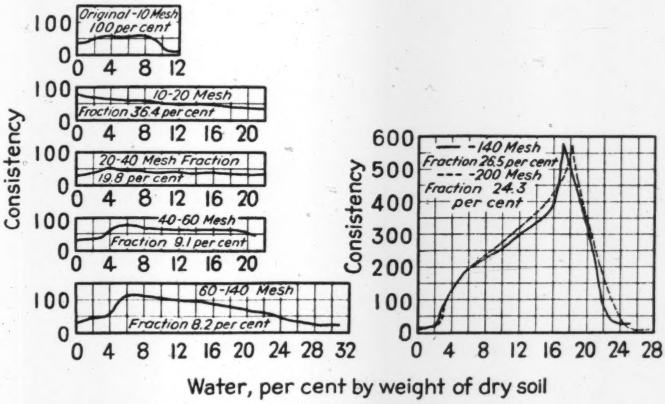

FIG. 10.—Plastograph Curves for South Carolina Topsoil (8-213) and Fractions.

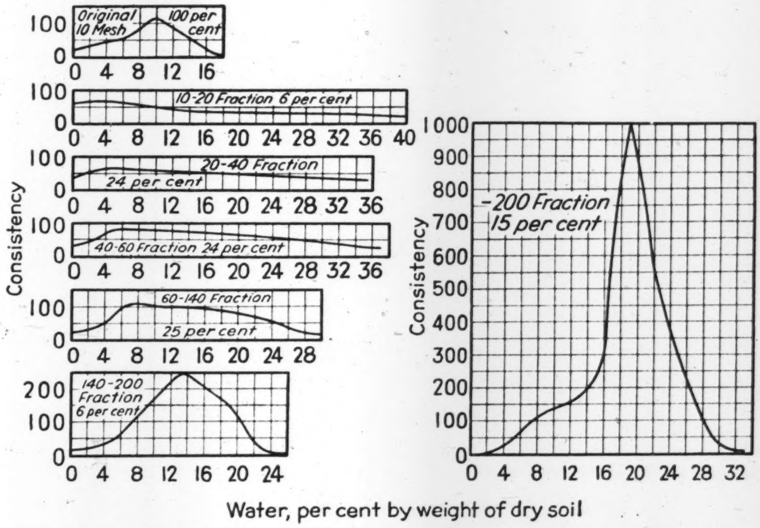

FIG. 11.—Plastograph Curves for Georgia Topsoil (8-152) and Fractions.

The 20 to 40, 40 to 60, and 60 to 140 mesh fractions had lower initial consistencies than the 10 to 20 mesh fractions, due probably to less friction between the smaller particles; but the consistency of each of these fractions, unlike the 10 to 20 mesh fraction, rose to a maximum when water was added and then decreased again to a minimum. The maximum consistencies reached and the amounts of water required to obtain maximum consistency were in reverse order to the average sizes of the particles in the different fractions.

The −140 mesh fraction had a lower initial consistency than any of the others, required more water to reach maximum consistency and its consistency at the maximum was much greater than that of any of the other fractions.

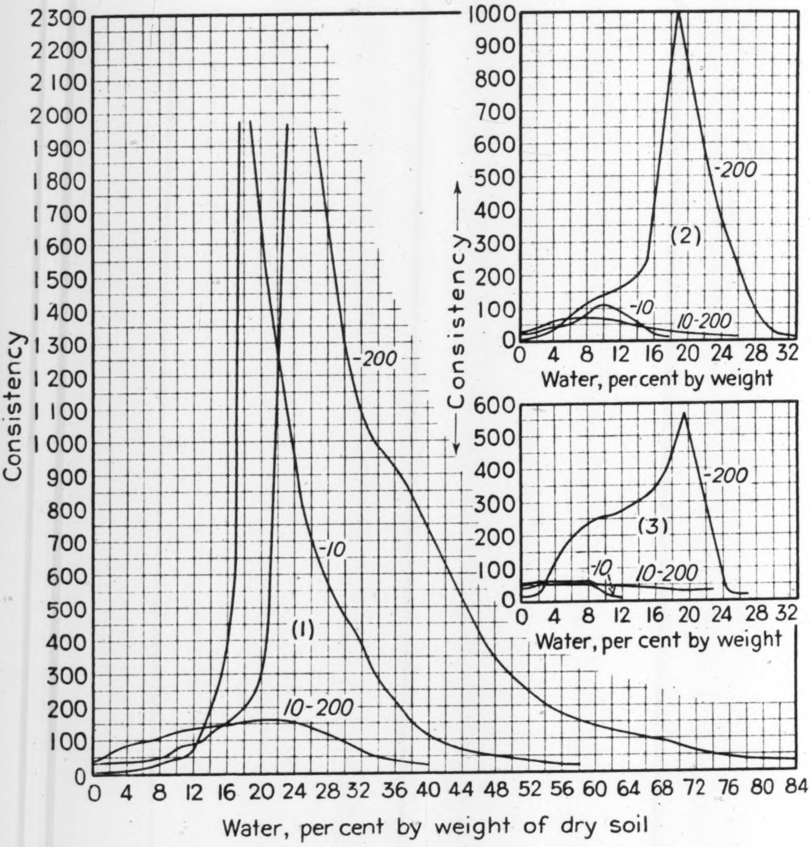

FIG. 12.—Plastograph Curves for 10 mesh (1) North Carolina Clay Soil (8-333), (2) Georgia Topsoil (8-152), (3) South Carolina Topsoil (8-213) and Their 10−200 and −200 Fractions.

The curve for the −200 mesh fraction parallels the curve for the −140 mesh fraction very closely, probably because it comprised 87 per cent of the latter.

The curves show plainly that the water-absorbing capacity of soil *A* (good topsoil from South Carolina) is confined principally to the −140 mesh fraction and possibly even to the −200 mesh fraction.

The curve for the topsoil as a whole (original −10 mesh) which is known to be stable in the presence of water, shows that the relatively great activity of the −140 mesh fraction (or −200 mesh fraction) was largely offset (stabilized) by the lower activities of all of the other fractions.

Investigation No. 2—Fig. 11:

In order to find out whether the same sieve fractions from different soils are affected similarly by water and to determine whether the activity of a −140 mesh soil fraction is due largely to its −200 mesh portion, we ran plastograph tests on a sample of topsoil from Georgia (said to be reasonably satisfactory) and on its various fractions and compared the plastograph curves shown in Fig. 11 with those obtained for soil *A*.

This comparison showed that the 10 to 20, 20 to 40, 40 to 60, and 60 to 140 mesh fractions were affected similarly and to about the same extent in both cases.

The 140 to 200 mesh fraction from the Georgia topsoil was affected somewhat more than any of the above fractions but by far the greatest activity was exhibited by the −200 mesh fraction. Furthermore, the activity of the −200 mesh fraction from the Georgia topsoil was much greater than that of the same fraction from South Carolina topsoil.

The greater activity of its −200 mesh fraction, although present in smaller amount, caused the Georgia topsoil as a whole (original −10 mesh, Fig. 11) to be affected more by water than the South Carolina topsoil (original −10 mesh, Fig. 10).

In general, the investigation showed that the individual fractions above −200 mesh from these two topsoils are affected similarly by water but their −200 mesh fractions behave very differently and these differences are imparted to the respective topsoils in proportion to the quantities of −200 mesh fraction present and the extent to which they are stabilized by the larger size fractions in the respective soils.

Investigation No. 3—Fig. 12:

To confirm the above conclusions and to find to what extent the amount and character of the −200 mesh fraction may influence a given soil, the two topsoils discussed above were compared with a very unstable clay soil (8-333) from North Carolina, in the following manner: Plastograph curves were prepared for the North Carolina sample as a whole (original −10 mesh) and for its 10 to 200 mesh and −200 mesh fractions. These curves are compared, in Fig. 12, with similar curves for Georgia topsoil (8-152) and South Carolina topsoil (8-213). The three sets of curves are designated as 1, 2 and 3, respectively. The sieve analyses for these three soils are given in Table II. Unlike the two comparatively stable topsoils, the very unstable North Carolina clay soil, as a whole, has a curve approximating in shape and size the curve for its −200 mesh fraction. The reasons are obvious. Its −200 mesh fraction, which amounts to 70 per cent of the total soil, has such great activity that it almost completely offsets the stabilizing effect of the 10 to 200 mesh fraction. Furthermore, the latter consists principally of 60 to 200 mesh material and, therefore, has a smaller stabilizing capacity than the 10 to 200 mesh fractions from either of the topsoils, which contain appreciable amounts of 10 to 60 mesh particles.

TABLE II.—SIEVE ANALYSES OF SOILS 1, 2, AND 3.

	Soil 1 (8–333) North Carolina Clay Soil	Soil 2 (8–152) Georgia Topsoil	Soil 3 (8–213) South Carolina Topsoil
Sieve analysis:			
Percentage passing No. 10 sieve..	100	100	100
" " " 20 " ..	99	64	94
" " " 40 " ..	97	44	70
" " " 60 " ..	95	35	46
" " " 140 " ..	84	27	21
" " " 200 " ..	70	24	15

Investigation No. 4—Figs. 13 and 14:

Having found from the preceding investigations that the water-absorbing capacity of any given soil is determined largely by the amount and character of its −200 mesh fraction, we wished to

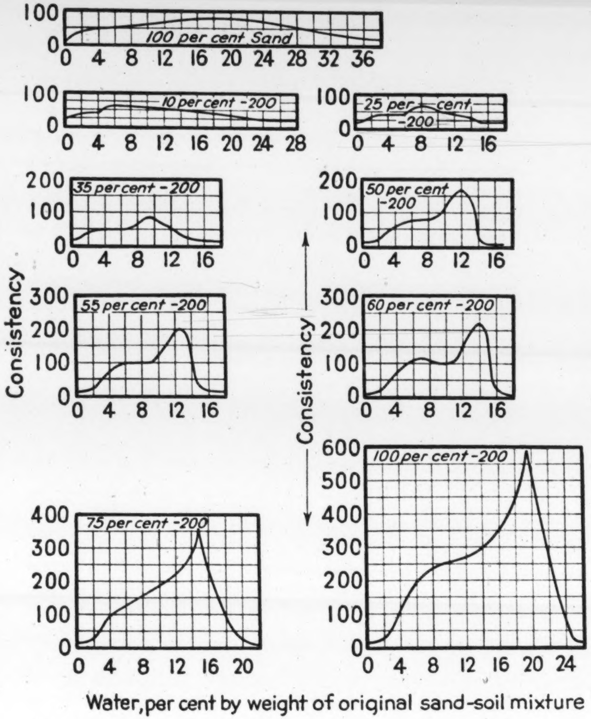

FIG. 13.—Plastograph Curves for Mixtures of 40 to 140 mesh Sand and −200 mesh Fraction from South Carolina Topsoil (8–213).

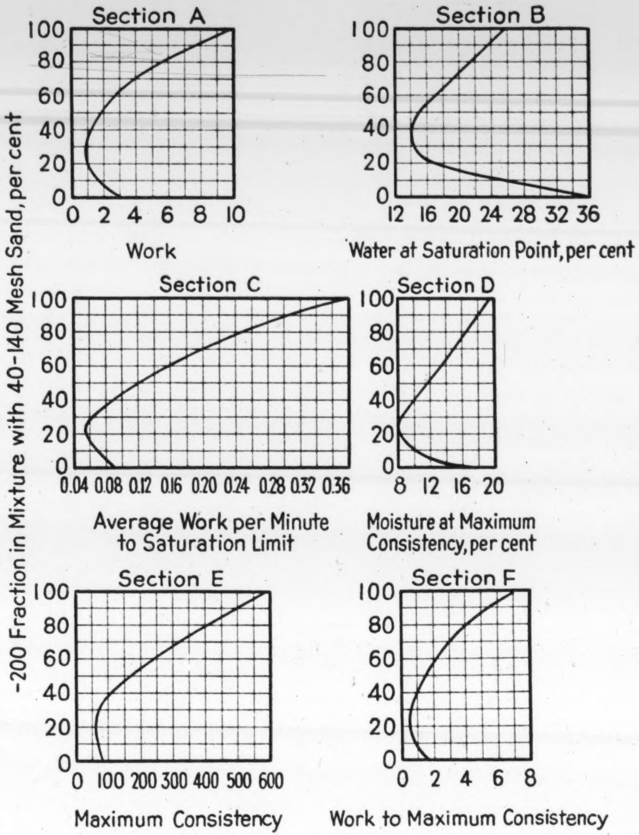

FIG. 14.—Influences of Increasing Amounts of −200 mesh Fraction from South Carolina Topsoil on Mixtures Containing 40 to 140 mesh Sand as Shown by Plastograph Curves.

determine whether variations in the amount of this fraction affect the total soil in some regular manner. For this investigation we mixed 40 to 140 mesh sand with the −200 mesh fraction from South Carolina topsoil (soil *A*, 8-213) in the following proportions:

SAND, PER CENT	−200 MESH FRACTION, PER CENT
100	0
90	10
75	25
65	35
50	50
45	55
40	60
25	75
0	100

From some preceding tests we had found that the mixture of 45 per cent sand and 55 per cent −200 mesh fraction from South Carolina topsoil duplicated the −40 mesh fraction from South Carolina topsoil amost exactly when tested with the plastograph. This close similarity to the natural product seemed to warrant the use of our two component synthetic mixtures for the purposes of this investigation. The replotted plastograph curves for this series of tests are shown in Fig. 13. It is evident from the gradual change in shape from 100 per cent sand to 100 per cent −200 mesh soil that changes in percentage composition shift the various significant parts of the curves in some regular manner. This is clearly shown by Fig. 14, the various sections of which present different relationships. For example, the area under each curve in Fig. 13 to the "saturation limit" represents the total amount of work performed by the plastograph during the addition of the water. This area, which represents gram meter minutes, may be measured with a planimeter and plotted, either in square inches, as in section *A*, Fig. 14, or in gram meter minutes, or other convenient work units, against percentage of −200 mesh soil in the mixture. When this was done the curve shown in Fig. 14, section *A*, was obtained. For section *C* (Fig. 14) the total work used in section *A* was divided by the total number of minutes required to perform that amount of work. The result, which the curve represents, is average work per minute. Both sections *A* and *C* show that the amount of work performed is least when 75 per cent sand is mixed with 25 per cent −200 mesh soil. We believe this may be explained as follows: The friction between the particles of sand is decreased by additions of −200 mesh soil, which serves somewhat as a lubricant, until 25 per cent of the latter is present. However, with further additions, the high consistency which the −200 mesh soil develops by itself in the presence of water commences to offset the lubricating action of smaller amounts and the work expended by the plastograph increases regularly to a maximum at 100 per cent −200 mesh soil.

Thinking that the work expended to the point of maximum consistency might be especially significant, the curve shown in section *F* (Fig. 14) was plotted. The minimum point on the curve was again at 75 per cent sand, 25 per cent −200 mesh soil. The same result was obtained when percentage of −200 mesh soil was plotted against percentage of moisture at maximum consistency (section *D*, Fig. 14) and percentage of −200 mesh soil against maximum consistency (section *E*, Fig. 14), but the result for percentage of −200 mesh soil plotted against percentage of water at "saturation limit" gave a minimum of 35 per cent −200 mesh soil, instead of 25 per cent as in all of the other cases. This one exception probably was due to the fact that the position of the "saturation limit" is not as sharply defined, especially in the coarser mixtures, as the other distinguishing points, that is, work area,

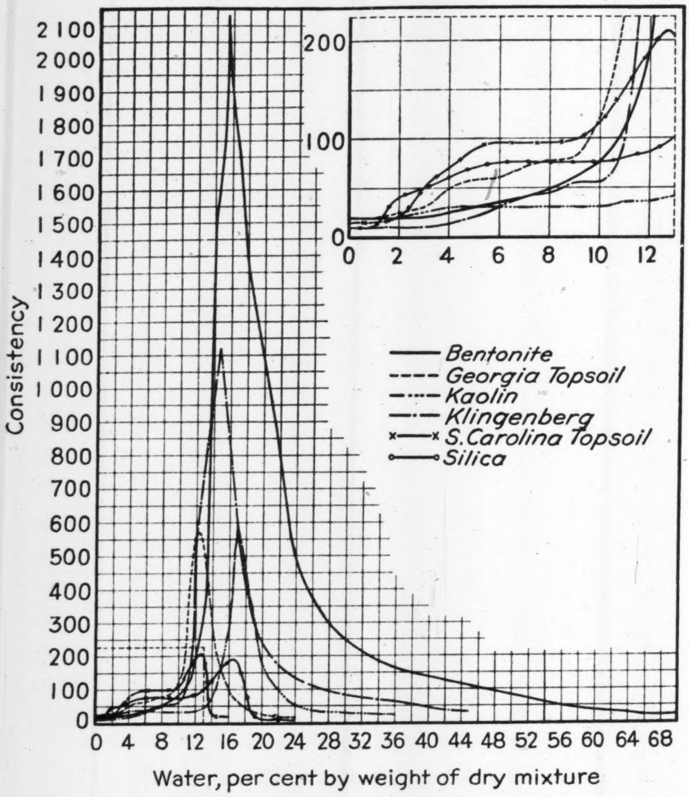

FIG. 15. Plastograph Curves for Mixtures of 40 per cent Sand (40 to 140 mesh) and 55 per cent of Various −200 mesh Materials.

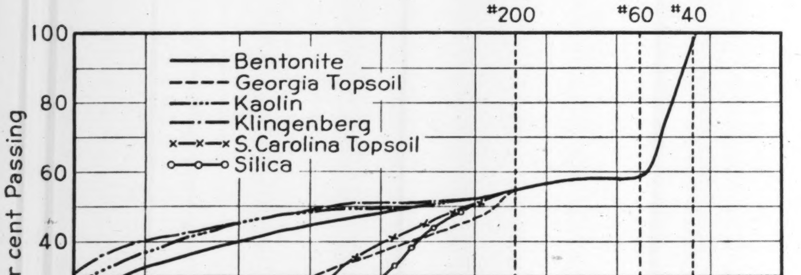

FIG. 16.—Gradation Curve for 40 to 140 mesh Sand and Bentonite, Georgia Topsoil, Kaolin, Klingenberg, South Carolina Topsoil and Silica.

maximum consistency and water content at point of maximum consistency.

We have arrived at the following conclusions as the result of this study: When a given −200 mesh soil is mixed in varying proportions with a given 40 to 140 mesh sand the total or average work to saturation limit, total work to maximum consistency, magnitude of maximum consistency, percentage of water to maximum consistency and probably the percentage of water to the saturation limit all decrease from 100 per cent sand to a minimum (75 per cent sand in this case) and then increase to a maximum at 100 per cent −200 mesh soil. The possibility of using the plastograph to advantage in determining the optimum amount of fine sand required to stabilize a fine soil is obvious.

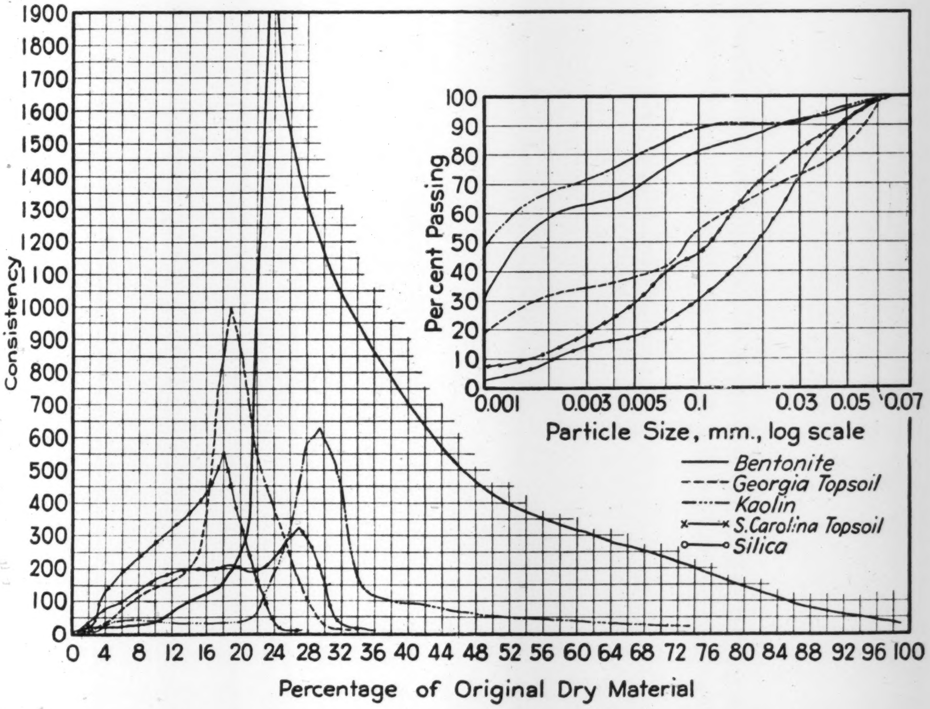

FIG. 17.—Plastograph and Size Gradation Curves for Various −200 mesh Materials.

Investigation No. 5—Figs. 15 and 16:

For the curves in Figs. 13 and 14 only one −200 mesh soil was mixed with the 40 to 140 mesh sand, but we know from experience and from our previous plastograph studies that different −200 mesh materials have different water-absorbing capacities, and should give different results when mixed with the same amount of a given sand. To check this point we mixed 45 per cent of the same 40 to 140 mesh sand used in the previous study with 55 per cent of each of the following −200 mesh materials: silica, −200 mesh fraction from South Carolina topsoil, −200 mesh fraction from Georgia topsoil, kaolin, Klingenberg clay and Bentonite. The replotted plastograph curves are shown in Fig. 15 The cumulative grain size curves for these same mixtures are presented in Fig. 16. In comparing these curves it should be remembered that each mixture contained the same amount of the same sand. This accounts for the fact that all of the cumulative grain size curves join to form a single curve at 55 per cent −200 mesh soil or No. 200 sieve. Obviously, the great differences in the plastograph curves are due solely to differences in the characters of the −200 mesh materials, since their quantities were the same. That these differences were not due, at least entirely, to difference in gradation is shown by the following comparison in which the mixtures are arranged according to desirability as indicated by gradation curves and as indicated by the plastograph:

	GRADATION CURVES	PLASTOGRAPH CURVES
1	Silica	South Carolina topsoil
2	South Carolina topsoil	Silica
3	Georgia topsoil	Georgia topsoil
4	Bentonite	Kaolin
5	Kaolin	Klingenberg
6	Klingenberg	Bentonite

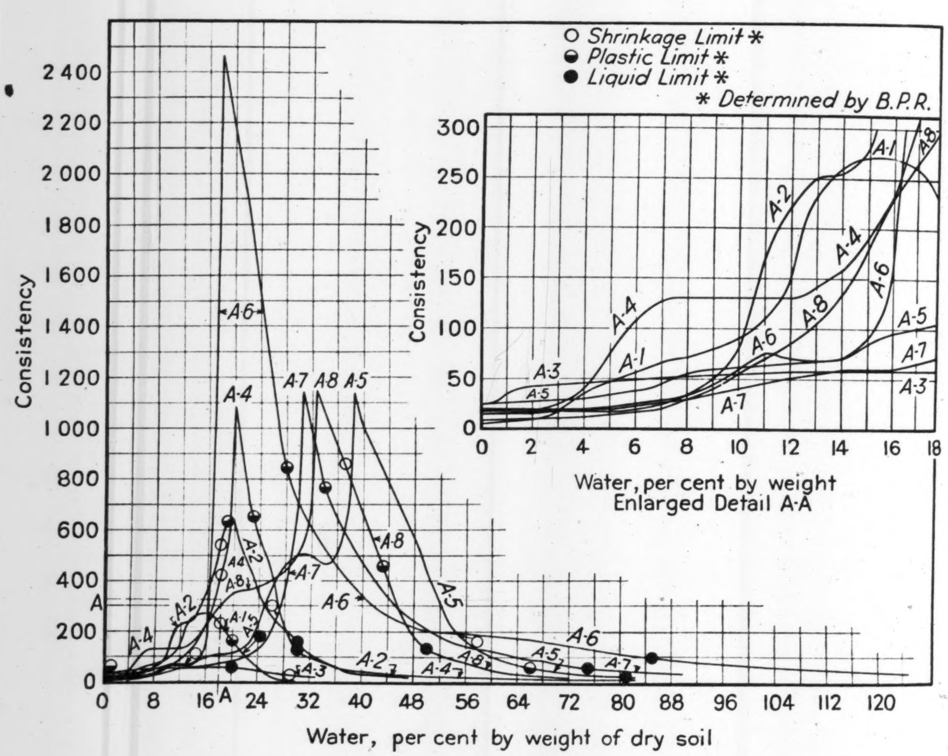

FIG. 18.—Plastograph Curves for 8 Bureau of Public Roads Standard Soils (−40 mesh) Shrinkage Limit, Plastic Limit, Liquid Limit.

Obviously soil gradation curves take into account only particle size and this alone does not determine the water-absorbing capacity of a −200 mesh soil. On the other hand, the plastograph curve undoubtedly presents a picture of the combined effects of particle size and aggregation, and all the other differences such as shape-size distribution, chemical composition, surface layer activity and the thickness and character of the water films on the soil particles which are supposed to affect the behavior of fine soils in the presence of water. In our studies of soil stabilization it is this combined effect which concerns us most and which we wish to measure. However, if the individual effects need to be known they undoubtedly can be determined with the plastograph by changing the different variables individually and measuring the effect of each.

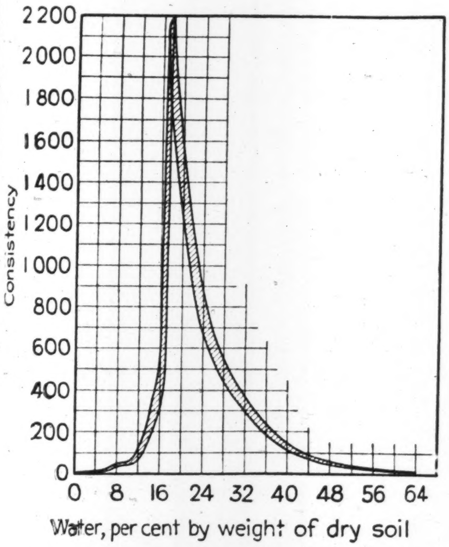

FIG. 19.—Band Enclosing Nine Separate Plastograph Curves for North Carolina Clay Soil (8–333) to Show Reproducibility of Plastograph Results.

Investigation No. 6—Fig. 17:

In this investigation each of the −200 mesh materials used in the series discussed above was tested with the plastograph without adding sand. The curves resulting from these tests are shown in Fig. 17, together with the cumulative grain size curves for each. They are similar in all respects to the curves in Fig. 15 but are larger because no comparatively inactive sand was added to dilute the active −200 mesh materials. In their undiluted form, the −200 mesh materials require more time and somewhat more care than their mixtures with sand and, considering the fact that the sand mixtures vary in the same order as the undiluted materials, it is possible and somewhat more desirable to use diluted mixtures for studies of this kind. This suggests the possibility of rating each −200 mesh material on the basis of the percentage required to impart a certain consistency to some standard sand. This would be a convenient index number that should have a practical significance.

Investigation No. 7—Fig. 18:

Our plastograph studies have indicated that the point on a plastograph curve at which an abrupt increase in consistency begins should correspond to the moisture content above which shrinkage occurs on drying; that the point of maximum consistency should correspond to the critical moisture content or plastic limit and that the point at which the plastograph curve changes direction for the last time and approaches a horizontal position should represent a liquid limit.

In order to check these assumptions a set of typical soil samples furnished by the Bureau of Public Roads was tested with the plastograph. The shrinkage limits, plastic limits, and liquid limits reported by the Bureau of Public Roads were then located on the plastograph curves as shown in Fig. 18. Inspection of Fig. 18 shows that there was no good agreement between the locations of the shrinkage limits and the points at which abrupt increases in consistency began. The same was true of the plastic limits with respect to the points of maximum consistency. In fact the locations of the shrinkage limits and plastic limits were not confined to any one section of the different plastograph curves and did not always occur in the same order with respect to each other. The liquid limits were somewhat better in these respects. They were located on approximately the same sections of the plastograph curves and coincided, with a fair degree of accuracy, with those points at which the curves changed direction for the last time.

Approximately the same results were obtained when plastograph curves for thirteen −40 mesh soil samples were compared with the shrinkage, plastic, and liquid limits for the same soils. As in the case of the Bureau of Public Roads soils there was fair agreement between the A.S.T.M. liquid limits and those estimated from the plastograph curves, but poor agreement between the shrinkage and plastic limits and points estimated from plastograph curves.

This lack of agreement between the significant points on plastograph curves and A.S.T.M. soil constants seems to indicate that there is poor reproducibility of either or both, or that the A.S.T.M. constants do not correctly indicate points at which the changes in state occur in soil-water mixtures.

That A.S.T.M. soil constants are not reproducible with a high degree of accuracy, especially when performed by different operators, has already been mentioned, but the extreme variations between the significant plastograph points and soil constants, at least in cases of the shrinkage and plastic limits, seem to indicate that those constants are not equally reliable or significant when different types of soil are tested.

Investigation No. 8—Fig. 19:

In the discussion of investigation No. 7, reference was made to possible poor reproducibility of plastograph curves. Actually we have found that the curve for any given soil may be reproduced very accurately. All of the tests performed in Germany were checked by duplicate runs and the two curves for each soil were practically identical in every case.

Recently a sample of 40-mesh North Carolina clay soil (8-333) was tested nine times with the plastograph. The curves all fell within the narrow shaded band shown in Fig. 19. Especially significant was the fact that the moisture contents at the more significant points varied only slightly in the different tests and, if these points represent the boundaries of different soil states, as we believe they do, the accuracy with which they may be reproduced should make it possible to use them for soil evaluation purposes in place of soil constants determined by present methods.

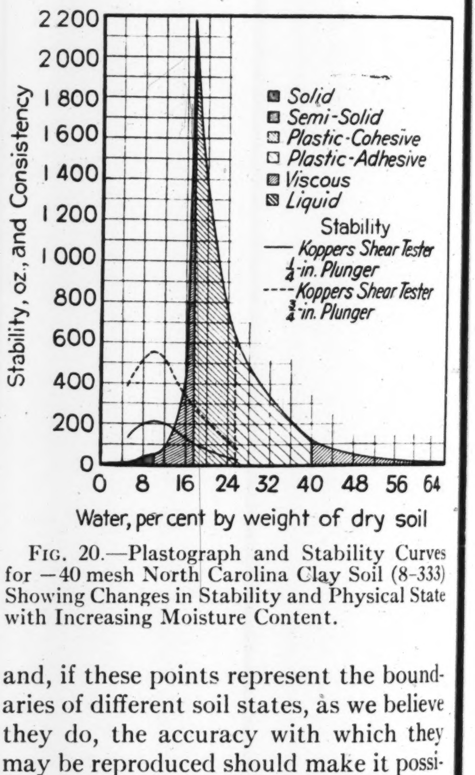

FIG. 20.—Plastograph and Stability Curves for −40 mesh North Carolina Clay Soil (8-333) Showing Changes in Stability and Physical State with Increasing Moisture Content.

Investigation No. 9—Fig. 20:

We have referred above to our belief that the more significant points on the plastograph curves may define the boundaries between different soil states. In Fig. 20 is shown an average plastograph curve for the nine separate tests on one soil (8-333) which were mentioned in the previous investigation. The area under this curve is divided into sections, each of which is intended to represent a different soil state. In the following paragraphs we have attempted to explain the significance of each section.

Section 1—Solid State—0 to 10 per cent Water.—As water is added to the soil, it first wets the surfaces of the particles (of all sizes and degree of activity) with which it comes in contact. Since much of this material may presumably be colloidally inactive, little increase in consistency is to be expected, especially in view of the short time of contact between the two phases. Throughout this section the soil-water mixture behaves like a granular material. The maximum limit of the section is the point or moisture content at which the curve starts to rise abruptly. It probably is the maximum amount of water which the soil can contain and not shrink appreciably on drying.

Section 2—Semi-Solid State—10 to 17.5 per cent Water.—Additional water beyond the maximum limit of section 1 probably fills the capillaries and penetrates between the layers of the soil particles causing swelling, absorption, or gelation to occur. This causes a rapid rise in consistency which continues until all the (exposed) colloidal material has reacted with the water to enter a gelatinous state and a point of maximum consistency is reached. This appears to be the true plastic limit.

Section 3—Plastic State (17.5 to 25.5 per cent Cohesive Plastic, 25.5 to 40 per cent Adhesive Plastic).—As more water is added, the gel formed in section 2 probably is diluted but remains in the gel state and the mixture as a whole remains plastic. The dilution of the gel causes the consistency of the mixture to drop more or less abruptly, depending upon the quantity and character of the gel formed during the preceding period.

This section of the curve appears to divide itself into two parts when highly plastic clays are tested. The upper portion of this section represents a cohesive but non-sticky plastic state which changes in the lower portion to a sticky, adhesive, plastic state. In the latter condition, the soil-water mixture adheres to all surfaces with which it comes in contact. This changes more or less abruptly to a viscous state represented by section 4. The water content at this point is assumed for the present to represent the transition point from the plastic state to a viscous liquid state and to represent a liquid limit.

Sections 4 and 5—Viscous and Liquid States—40 per cent Plus.—Section 4 in which the soil-water mixture is in a viscous state should, with the addition of sufficient water, extend to the flocculation limit beyond which the soil is in a liquid state (section 5). Thus far no attempt has been made to locate the flocculation limit by plastograph curves.

Investigation No. 10—Fig. 20:

Thus far our discussions of plastograph curves have dealt only with moisture-consistency relationships. We have attempted to show that the plastograph can be used, apparently with a high degree of accuracy, to determine whether and to what extent any given fine soil or soil fraction becomes plastic when mixed with water. It also can be used to determine the effects of admixtures, again in terms of moisture-consistency relationships or, if preferred, in terms of moisture contents at certain significant points on the consistency curves corresponding to shrinkage limit, plastic limit and liquid limit.

Considering the fact that a topsoil that is known to be highly stable in service when wet has a short and substantially flat plastograph curve and that a clay soil known to be unstable in service when wet has a long and irregular plastograph curve, it seems evident that the plastograph indicates the instability of soils with or without admixtures, when they are mixed with water. Those having the greatest areas below their plastograph curves are the least stable in service. Comparing soils on the basis of plastograph curves is similar to comparing them on the basis of plasticity indices with the difference, we believe, that the plastograph comparisons are more accurate and they can be applied to soils containing bituminous admixtures, whereas plasticity indices cannot. This last fact has resulted in the use of stability tests, usually of the shear type, to determine the relative stabilities of soils containing bituminous admixtures after contact with or immersion in water.

Believing that there must be some relationship between stability as measured by shear and instability as indicated by plastograph curves, we mixed portions of the same soil that was used for Fig. 20 (8-333) with various amounts of water, compacted 8-g. portions (based on dry soil) in 1-in. molds at 100 lb. total pressure and then tested them immediately for shear with the Koppers shear tester (Figs. 1 and 2) using both $\frac{1}{4}$- and $\frac{3}{4}$-in. plungers. The stabilities in ounces were plotted against moisture content (on the basis of dry soil), determined by oven drying. The curves obtained in this manner are included in Fig. 20. They indicate that, with increasing amounts of water, the stabilities both with the $\frac{1}{4}$-in. and with the $\frac{3}{4}$-in. plunger rose to a maximum at 10 per cent water content, which we had selected as the limit of the solid state, then decreased slowly through the semi-solid and cohesive plastic state, reaching minimum values in the adhesive-plastic state. The same comparisons were made with three other soils with the same results, which leads us to believe that each soil has a maximum stability, as measured by static shear resistance, at that point or moisture content where the semi-solid state begins. After that point is passed, any stability due to friction and cohesion is overcome by the lubricating and diluting effects of the increasing water content.

Another indication from the stability testing, which is not shown in Fig. 20, is that clay soils whose service behaviors are known to be unsatisfactory have higher shear values at the points of maximum stability than those soils that are known to have good service behaviors. In other words, the static shear test results and known service results appear to be in reverse order. On the other hand, the plastograph curves portray the instabilities of the different soils in proper order.

This important phase of the subject deserves and will receive further study, but at present it appears that comparisons of instabilities with the plastograph are more reliable than comparisons of stabilities with shear tests. This leads us once more to conclude that shear tests are unsatisfactory because they are static tests which do not include motion like that encountered under service conditions.

CONCLUSION

From these preliminary studies we have concluded that the use of the plastograph will enable us to conduct soil studies more intelligently and predict with greater certainty the behaviors of soils which are to be used for highway purposes with or without admixtures of stabilizing agents.

Acknowledgment:

The author wishes to express grateful acknowledgment of assistance received from his associates in the Koppers Co., Tar and Chemical Division, and from Mr. Brabender and members of his staff which made possible the presentation of this report of plastograph investigations.

DISCUSSION

Mr. Donald M. Burmister.[1]—The behavior of soil represents the interaction and the influence of a great many physical characteristics. Our difficulty lies in separating the effects of the different physical factors and correlating behavior with one, two or three prominent physical characteristics as a means of predicting behavior. It seems as though the curves obtained by Mr. Rhodes have rather definite critical points, although I do not know just what they mean. There should be a great deal more study given to the curves in order to interpret just what significance these critical points have. I would not go so far as to say that the liquid, plastic, and shrinkage limit were not of equal or even greater significance. It may be that they represent quite a different phenomenon from that developed in the new test. However, I do believe that the liquid and plastic limits, particularly, are influenced by the same physical factors that determine stability and shear strength. Just what the relations are in the case of the new test are not clear. I cannot quite see why there should be any definite relation of the liquid, plastic, and shrinkage limits to these critical points on the curves. It seems as though this phenomenon has something to do first with the wetting properties, and the capillary tendencies of the soil up to the maximum point. At first, the moisture only wets the individual grains but they do not adhere. Where they begin to adhere and form a cohesive mass, there is a sharp rise in the curve. The soil then becomes more and more cohesive and, in plastic soils, a more sticky mass. At the maximum point, the greatest effort is required to mix the material. This corresponds, in a way, to an optimum moisture condition, somewhat similar to that obtained in the compaction tests. Thereafter, the addition of moisture appears to cause a swelling or softening of the material, and the effort required to mix the sample drops rapidly. A condition is finally reached at which the mixing effort remains almost constant, for a semi-liquid state.

There are two factors of importance in the test: first, the moisture content at which the peak of the curve comes; and second, the magnitude of this peak as a relative measure of mixing effort.

The most important work to be done is: first, to determine the physical significance of the test and to define the physical factors involved; and second, to continue the correlation of these physical characteristics of soils with their actual performance as subgrade soils under various circumstances, as a means of classifying subgrade materials for use.

Mr. Raymond Harsch.[2]—I should like to ask in connection with the use of this machine with bituminous materials, would it not be necessary that the materials be considered after treatment to be in a pulverized state rather than cemented together?

Mr. E. O. Rhodes.[3]—Up to the present time the tests have been conducted only on dried and pulverized soils or soil mixtures. We have been most concerned with the study of the significance of the results obtained and their interpretation in terms of known soil characteristics. We intend to carry out experiments to determine whether significant tests can be run on soil samples as they come from the road without necessity of subjecting them to the preliminary drying, since it is possible that important changes in the properties of treated soils may be brought about by this drying.

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[1] Assistant Professor of Civil Engineering, Columbia University, New York City.

[2] Assistant Manager, Asphalt Dept., Shell Oil Co., San Francisco, Calif.

[3] Technical Director, Tar and Chemical Division, Koppers Co., Pittsburgh, Pa.

We are also considering the possibility of testing samples representing the entire soil passing a 1-in. screen instead of testing a portion of the soil as we do at present. In this connection we have discussed with Mr. Brabender the possibility of building a larger and more powerful plastograph. Plans for the construction of such a device are being considered at the present time.

A STUDY OF DEFLOCCULATING AGENTS USED IN THE PARTICLE SIZE DETERMINATION OF SOILS

By Edward E. Bauer[1]

In the determination of the sizes of particles of soil, as outlined in the Society's Tentative Method of Mechanical Analysis of Soils (D 422 – 35 T)[2] it is specified that 20 ml. of sodium silicate, having a specific gravity of 1.023 at 67 F. (19.4 C), be used as a dispersing or deflocculating agent. In this method of test the quantity of suspension prepared is 1 liter. The U. S. Bureau of Public Roads reports in an article by Thoreen,[3] that "extensive tests with various deflocculating agents showed sodium silicate to be the most effective for maintaining separation of the dispersed soil particles in suspension." Thoreen indicates that other deflocculation agents included were potassium hydroxide, ammonium hydroxide, sodium carbonate and sodium oxalate. The amounts of the various agents used were quite small.

The determination of sizes of particles of soil by a sedimentation method is based on Stokes' law, which in turn is based on a single particle falling in a liquid indefinite in extent. In the tentative method of test 50 g. of clay soil or 100 g. of sandy soil are used per liter of suspension. Casagrande reports that for the above amounts, the concentration is not sufficient to invalidate Stokes' law.[4] Each particle of soil must, however, be entirely separated from every other particle by water. Most of our soils flocculate when placed in water—that is, particles gather themselves together in clusters. These clusters have an entirely different settling velocity than the individual particles would have, and, therefore, make any determination of particle sizes by a sedimentation process impossible, unless something can be done to prevent flocculation.

The author found that various agents other than sodium silicate were in use because the sodium silicate was not functioning satisfactorily. As a result of this, the author began a study of various phases of the whole problem of deflocculation and the findings so far are presented herein. In view of the fact that many laboratories follow the A.S.T.M. procedure in the making of this test, it seems important to point out at this time some of the difficulties involved.

Pioneer Tests:

It was suggested to the author that the amount of the deflocculating agent that is added is a very important item. If there is too small an amount or too large an amount of agent present, flocculation will take place. It appears that the correct amount can be determined only by a cut-and-try process.

In order to determine the kind and correct amount of deflocculating agent to use with each sample of soil, the author made use of small quantities of suspension in test tubes $\frac{7}{8}$ in. in diameter and 10 in. long. Three grams of air-dried soil were used in a total suspension of 60 ml. This is almost exactly the same quantity of soil per liter of suspension as is used in the regular hydrometer test. The soil was weighed on a 3-beam balance having a capacity of 100 g. and with the smallest graduation 0.01 g. Various amounts of deflocculating agents were used in the different test tubes.

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[1] Assistant Professor of Civil Engineering, University of Illinois, Urbana, Ill.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 953 (1935); also 1937 Book of A.S.T.M. Tentative Standards, p. 885.

[3] R. C. Thoreen, "Comments on the Hydrometer Method of Mechanical Analysis," *Public Roads*, Vol. 14, No. 6, August, 1933, p. 95.

[4] A. Casagrande, "Die Araometer-Methode zur Bestimmung der Kornverteilung von Boden und anderen Materialen," p. 32, Julius Springer, Berlin (1934).

A holder was made so that 19 tubes could be inserted in a cylinder of the Deval abrasion machine. The samples were agitated for 1 hr., being thrown from one end to the other of the tube and back again during each revolution. Almost 2000 revolutions were run in 1 hr. The tubes were then placed in a rack in front of a window, in order to observe the sedimentation process. Observations were normally made up to 48 hr. on those tubes in which the suspension held up. In many instances the soil flocculated and settled out in a comparatively short time. The performance in the test tubes determined the amount of deflocculating agent to use in the hydrometer analysis.

In several cases it appeared as though the agitation in the test tubes was not equal to that of the regular mixer used in the hydrometer test procedure. For pioneer testing prior to the hydrometer analysis, it would be much better to use quantities that can be mixed with the regular mixer. Using a small special dispersion cup, the amount of the suspension could be kept between 100 and 200 ml.

Hydrometer Analysis:

For the hydrometer analysis a density type of hydrometer, having a range from 1.000 to 1.030, was used. Fifty grams of air-dried soil were used, except as noted in a few of the test reports. The soil was soaked first for a period of 18 hr., at the end of which time the deflocculating agent was added. Mixing was done with the mixer as specified in the A.S.T.M. procedure.[2] Density readings were taken at the following intervals, following the beginning of sedimentation: ½, 1, 2, 4, 8, 15, 30 min., 1, 2, 4, 8, 24, and 48 hr. This schedule was not rigidly observed whenever it was impossible to take the readings. Diameter values were computed for the actual times.

Diameters were computed from Stokes' formula:

$$D = \sqrt{\frac{30}{980}\,\frac{n}{G - G_1}\,\frac{L}{T}}$$

where n = viscosity in dyne seconds per square centimeter,

G = specific gravity of the soil particles,

G_1 = specific gravity of the water,

L = distance from the surface of the suspension to the level at which the hydrometer reading was taken, in centimeters, and

T = time from the beginning of sedimentation until the reading was taken, in minutes.

An effort was made to hold the temperature of the suspension at 20 C., which is the temperature at which this particular hydrometer was calibrated. Since the method of control was not automatic, small variations occurred in the temperature of the suspensions. Values of D have been computed assuming that the temperature remained constant at 20 C., there being no way of allowing for changes in temperature in the solution of the formula.

Percentages of soil remaining in suspension at the various time intervals, T, were computed from the formula:

$$P = \frac{100}{w} \frac{G}{G - G_1} (r'C + F)$$

where P = percentage of soil remaining in suspension at time, T,

w = weight of oven-dried soil per milliliter of suspension at the beginning of the test, in grams,

r' = density reading of the hydrometer minus 1.000 and with the meniscus correction applied (it is merely the decimal portion of the hydrometer reading), and

C and F = correction factors which are functions of temperature. When the temperature is in the vicinity of 20 C., the C factor does not change the value of r' an appreciable amount. When the temperature is 20 C., the factor F is zero.[5]

In view of the fact that we were interested only in the sedimentation process, no attempt was made to determine the sizes of the larger particles by a sieving method.

Dispersing Agents:

For the sodium silicate solution, water glass and distilled water were mixed to produce a solution having a specific gravity of 1.023 at 19.4 C.

Since a normal solution of sodium oxalate would require 67 g. per l. and it is soluble in water to the extent of 37 g. per l., it is obvious that a solution less than normal must be prepared. Since the half-normal solution is so close to the limit of solubility, a quarter-normal stock solution was prepared. All quantities of sodium oxalate mentioned in this report refer to a quarter-normal solution.

Soils Used:

A number of soils were tested from various parts of the United States. Judging from the results secured, they were of different mineralogical contents. No attempt has been made to determine the minerals present.

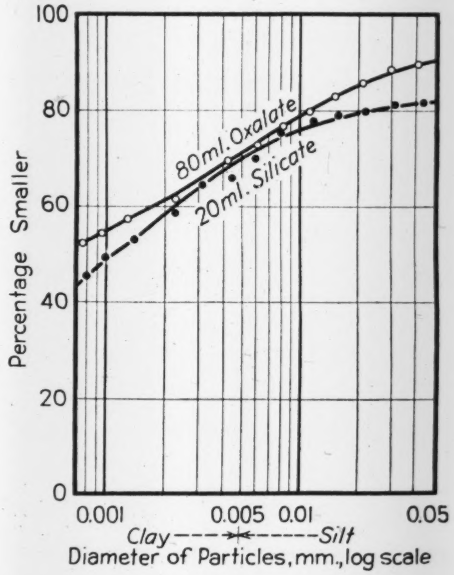

FIG. 1.—Soil No. 1. Comparison of Sodium Silicate and Sodium Oxalate as Deflocculating Agents.

Soil No. 1 is clay sample received from the Bureau of Public Roads several years ago.

Soil No. 2 is an Illinois soil furnished by the Illinois Geological Survey.

Soil No. 3 is an Illinois soil from near Peoria.

Soil No. 4 is from Oklahoma and was furnished by the soil-cement laboratory of the Portland Cement Association in Chicago. This soil had a decidedly red color.

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[5] E. E. Bauer, "Hydrometer Computations in Soil Studies Simplified," *Engineering News-Record*, Vol. 118, May 6, 1937, pp. 662–664.

Soil No. 5 is an Illinois soil from near Dixon.

Soil No. 6 is an Illinois soil, received from the Division of Highways, as part of a cooperative series of tests.

Soil No. 7 is another soil in same series as No. 6.

Soil No. 8 is a sample of silty soil from the Peoria, Ill., district of the Division of Highways.

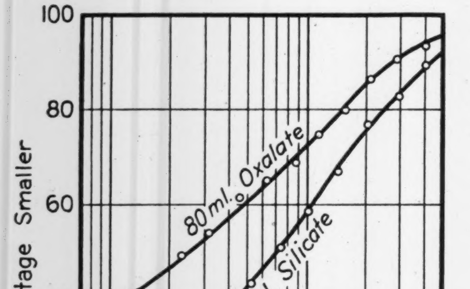

FIG. 2.—Soil No. 2. Comparison of Sodium Silicate and Sodium Oxalate as Deflocculating Agents.

Soil No. 9 is a foundation soil from Chicago. It was received from the Sanitary District.

Soil No. 10 is from the Dixon, Ill., district of the Division of Highways.

Comparison of Sodium Silicate and Sodium Oxalate:

Based on the performance in the test tubes, hydrometer analyses were made using the amounts of silicate or oxalate which appeared to give the best results. A brief description of the work with each soil is given.

Soil No. 1, Fig. 1.—The pioneer tests indicated that either sodium silicate or sodium oxalate was equally effective up to a period of several days. Hydrometer analyses were run on two samples of soil, in one of which 80 ml. of sodium oxalate was used and in the other 20 ml. of sodium silicate.

This test was carried out over an unusually long period of time. The oxalate appears to be the better dispersing agent, although at the 5 μ size, there is very little difference between the curves. Percentages of clay secured are: using oxalate, 71; using silicate, 69. Percentages of silt are: using oxalate, 19; using silicate, 13.

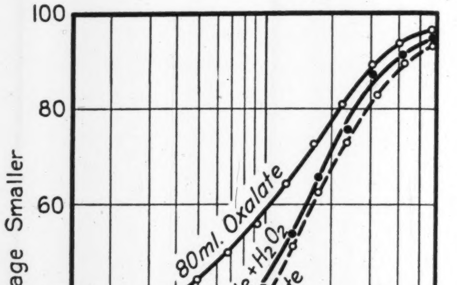

FIG. 3.—Soil No. 3. Comparison of Sodium Silicate and Sodium Oxalate as Deflocculating Agents.

In the case of the silicate, hydrogen peroxide was added to the suspension as recommended in the tentative method of test.

Soil No. 2, Fig. 2.—The pioneer tests showed that the oxalate was a better dispersing agent than the silicate. In a period of 15 hr., the sodium silicate samples were all fairly well settled out.

The tubes containing amounts of oxalate equal to 50 to 80 ml. per l. held up the best. In the hydrometer tests, 80 ml. of oxalate and 20 ml. of silicate were used.

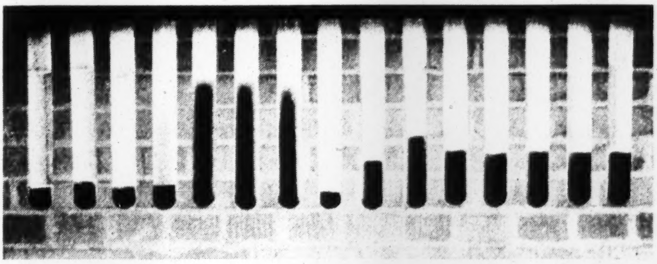

FIG. 4.—Soil No. 4. Pioneer Test Indications at the End of 22 hr.

The seven tubes at the left contain amounts of silicate varying from 16.7 to 66.7 ml. per liter of suspension; the seven at the right contain oxalate varying from 33 to 133 ml. per liter. The middle tube contains no deflocculating agent. The second one from the left contains silicate at the rate of 20 ml. per liter of suspension.

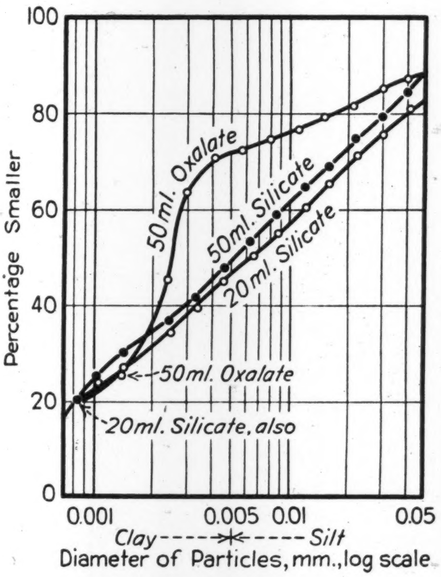

FIG. 5.—Soil No. 4. Comparison of Sodium Silicate and Sodium Oxalate.

The oxalate appeared to form a gel which accounts for the abnormal curves.

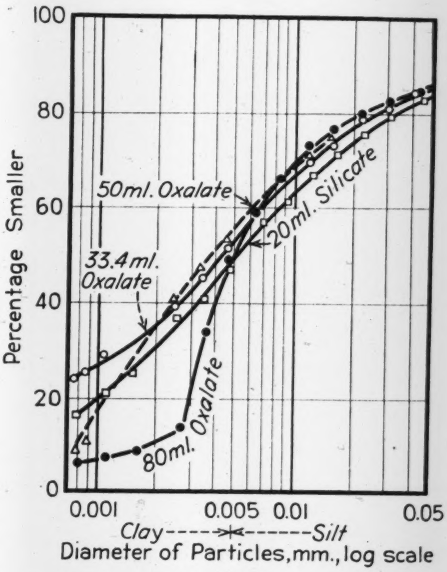

FIG. 6.—Soil No. 5. Comparison of 20 ml. of Sodium Silicate with Various Amounts of Sodium Oxalate.

33.4 ml. of oxalate was the best quantity tried, and is also better than 20 ml. of the silicate.

Percentages of clay obtained were: using oxalate, 61; using silicate, 42. Percentages of silt were: using oxalate, 34; using silicate, 50. There is a much larger difference here than in the case of soil No. 1, the difference being 19 per cent for clay and 16 per cent for silt.

Soil No. 3, Fig. 3.—Here again the pioneer tests indicated that the oxalate was better than the silicate and that the amount of the oxalate could vary over a wide range without affecting the results. Eighty milliliters of oxalate and 20 ml. of silicate were used.

The oxalate was the better dispersing agent, giving 45 per cent clay and 51 per cent silt, as compared to 24 per cent clay and 67 per cent silt for the silicate.

The tentative method calls for the use of hydrogen peroxide when the plasticity index is greater than 20. Since the value of the peroxide has been questioned, a check test was made on this sample of soil. The results indicated a slight difference in favor of the peroxide, although the amount is small and probably not greater than the experimental errors might be.

Soil No. 4, Figs. 4 and 5.—The pioneer tests in this case indicated that certain amounts of silicate were better than any amount of oxalate. From the test tubes it appeared that about 40 or 50 ml. of silicate per liter of suspension would give the best results. Hydrometer tests were run using 20, 33.4, and 50 ml. of silicate.

The sodium oxalate samples in the pioneer tests all settled out in an unusual manner. The sediment appeared jelly-like and the liquid above the sediment was clear. The level of the sediment became lower with time. Just to satisfy our curiosity a sample using 50 ml. of the oxalate was tested with the hydrometer, with the results shown in Fig. 5. There is not much difference between the results for the various amounts of the silicate. The curve for the 33.4 ml. has been omitted but it would lie between those for 20 and 50 ml. The curve for the 50 ml. of oxalate is abnormal and is of no value in determining particle sizes. Laboratories that carry the determination only far enough to get the 5μ size percentage might not pick up this peculiarity and not realize that they had an unusually high value.

Soil No. 5, Fig. 6.—The pioneer tests indicated that the oxalate was better than the silicate, but that a much smaller amount was better than the 80 ml. used in the previous tests. Hydrometer analyses were run using 16.7, 33.4, 50 and 80 ml. of oxalate and 20 ml. of silicate.

The results indicate that 33.4 ml. of the oxalate gave the greatest dispersion, although the curve for 16.7 ml. (omitted from the chart) is only slightly lower. The curves for the larger amounts of oxalate drop off rapidly for the smaller diameters. The 20 ml. of silicate did almost as well as the 33.4 ml. of oxalate, showing 49 per cent clay as against 53 per cent for the oxalate. The amount of the silt size is essentially the same for both agents.

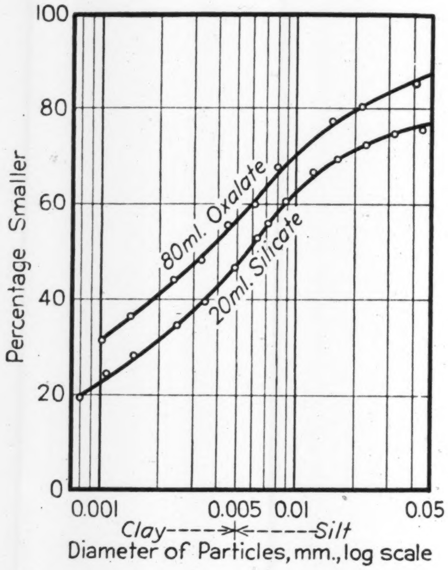

FIG. 7.—Soil No. 9. Comparison of Sodium Silicate and Sodium Oxalate as Deflocculating Agents.

This is a foundation soil from Chicago.

Soil No. 9, Fig. 7.—The pioneer tests indicated that the oxalate was better than the silicate, and hydrometer analyses were run using 80 ml. of oxalate and 20 ml. of silicate. These tests were run on air-dry soil. Later, mention will be made of tests using soil not dried.

Results show 56 per cent clay using the oxalate and 47 per cent with the silicate; the percentage of silt was 30.5 for both samples.

Soil No. 10, Figs. 8 and 9.—Here again the sodium oxalate gave the better results. In Fig. 8 is a picture of the tubes at the end of 22 hr. All the silicate samples have settled out and the smaller amounts of the oxalate are holding up.

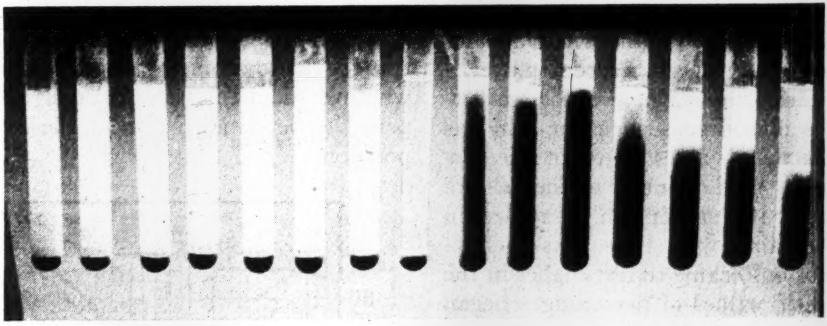

FIG. 8.—Soil No. 10. Pioneer Test Indications at 22 hr.

The seven tubes at the left contain silicate varying from 16.7 to 66.7 ml. per liter and the seven on the right contain oxalate in quantities varying from 33 to 133 ml. per liter of suspension. The middle tube contains no agent.

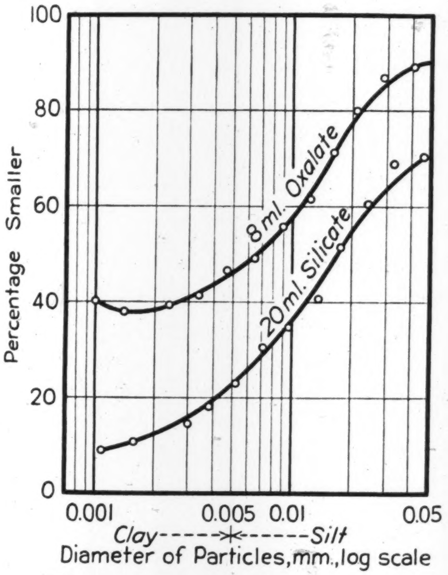

FIG. 9.—Soil No. 10. Comparison of 80 ml. of Oxalate with 20 ml. of Silicate.

In the hydrometer analyses only 80 ml. of oxalate and 20 ml. of silicate were tried. There is considerable difference in the results secured, the oxalate giving 46 per cent clay and 45 per cent silt, while the silicate showed 22 per cent clay and 49 per cent silt. The upturn of the oxalate curve may indicate the formation of a gel. This test was done

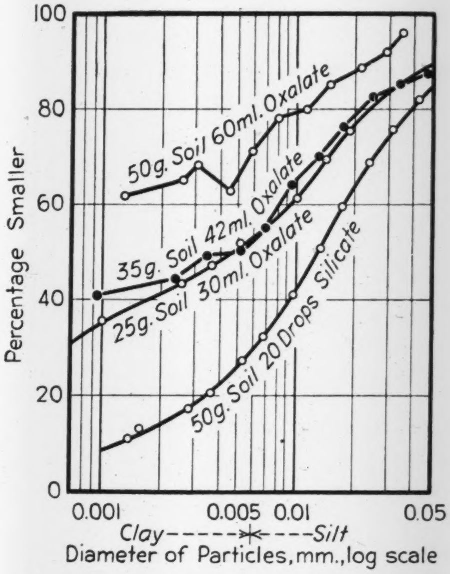

FIG. 10.—Soil No. 6. Comparison of the Results Secured with 50, 35, and 25 g. of Soil, Using Sodium Oxalate at the Rate of 60 ml. per 50 g. of Soil.

Result of an auxiliary test using 20 drops of sodium silicate instead of the usual 20 ml. is shown also.

early in the series and before the gel formations were so obvious in any of the suspensions.

Amount of Soil Dispersed:

When sodium oxalate was used with a number of soils a gel appeared to be formed and there was no settlement of particles of any size. Hydrometer readings taken in the gel gave very high percentages of soil in suspension. It was felt that these values were not the correct ones. The quantity of the gel-like portion of the suspension decreased in every instance with time. As the hydrometer bulb came to rest higher in the gel, erratic values of percentages began to appear.

Formation of the gel is easily detected since the liquid above the gel is clear, being free of all soil particles. It was interesting to note also that some of the gels were able to keep in suspension particles of sand sufficiently large to be easily discernible with the naked eye.

At the suggestion of J. E. Lamar, Geologist for the Illinois State Geological Survey, the amount of soil put in suspension was decreased until the suspension appeared to be normal. Hydrometer analyses up to 24 or 48 hr. seemed to give normal results. In several instances, however, readings beyond these time intervals indicate that a gel had finally formed. These densities were always higher than those that were considered normal.

Soil No. 6, Fig. 10.—The pioneer tests indicated that the silicate was not at all effective in holding the particles in suspension. When the oxalate was used, a gel appeared to form, using quantities of materials equivalent to 50 g. of soil per liter of suspension. Without trying smaller samples of soil in the test tubes, it was decided to try three quantities of soil in the hydrometer tests: 25, 35, and 50 g. The amount of oxalate used was at the rate of 60 ml. per 50 g. of soil.

The curve for 25 g. of soil and 30 ml. of oxalate seems to be the normal curve, whereas the other two using oxalate are highly erratic due to the fact that the gel had formed. The erratic nature of these curves is partly due to the fact that it is almost impossible to know when the hydrometer has come to rest. In some instances the hydrometer would remain at whatever depth it was placed if it happened to be somewhere near the correct position.

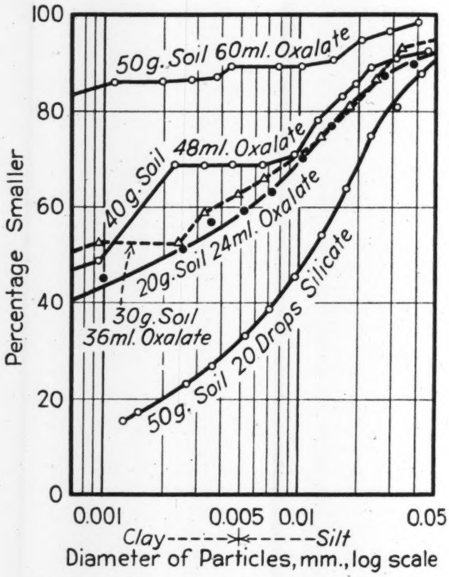

FIG. 11.—Soil No. 7. Comparison of the Results Using 20, 30, 40 and 50 g. of Soil per Liter, Using Sodium Oxalate at the Rate of 60 ml. per 50 g. of Soil.

Result of an auxiliary test using 20 drops of sodium silicate instead of the usual 20 ml. is shown also.

One suggestion made in connection with this whole project was that 20 ml. of sodium silicate is too large an amount and that quantity in the neighborhood of 20 drops would be much better. This amount of silicate was tried with soils Nos. 6 and 7, instead of the usual 20 ml. The curves for the 20 drops are shown in Figs. 10 and 11. It appears that for these two soils the effectiveness is not as great as is that of the oxalate.

Soil No. 7, Fig. 11.—Soil No. 7 acted much the same as did No. 6. Both were Illinois soils. In this case the amounts of soil used per liter of suspension were 20, 30, 40, and 50 g. and the amounts of oxalate were 24, 36, 48 and 60 ml.

From the curves shown in Fig. 11 it appears that 20 g. of soil is probably somewhere near the proper amount to use. There is no way of knowing, however, what the true curve is.

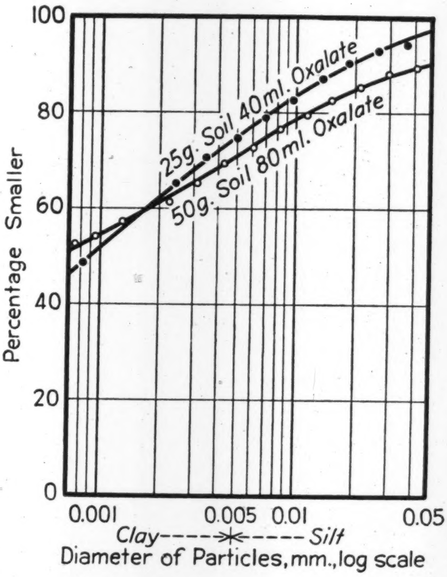

FIG. 12.—Soil No. 1. Comparison of the Results Using 50 and 25-g. Samples of Soil per Liter of Suspension.

The auxiliary test with 50 g. of soil and 20 drops of silicate gives a curve much lower than the one with 20 g. of soil and 24 ml. of oxalate.

Soil No. 1, Fig. 12.—After the experience with soils Nos. 6 and 7, another test was run on soil No. 1, using a 25-g. sample. The results are not much different and the difference may be due to the selection of samples, since they were not taken from the supply at the same time.

Effect of Drying the Sample:

Several samples were tested to see whether air drying of the soil might have any effect on the particle sizes.

Soil No. 8, Fig. 13.—Pioneer tests indicated that the oxalate was better than the silicate, so the comparison was made using 50 g. of soil and 80 ml. of oxalate in each case. The sample which was not dried first shows 26 per cent clay and 74 per cent silt, while the one that was air dried showed 19 per cent clay and 64 per cent silt.

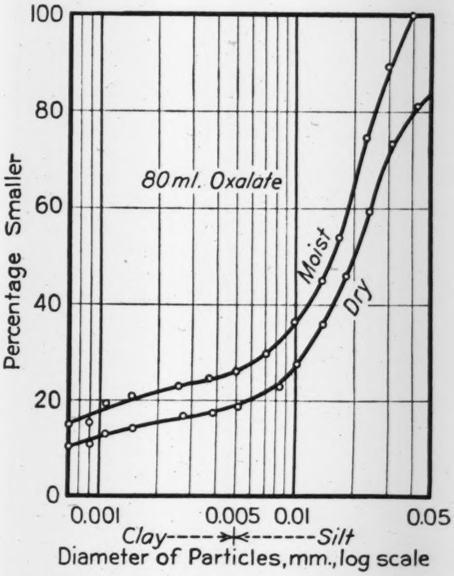

FIG. 13.—Soil No. 8. Comparison of an Air-Dried Sample with One That Had Its Original Moisture in It when Dispersed.

Soil No. 9, Figs. 14 and 15.—Pioneer tests on this soil indicated that the oxalate did a better job of dispersion than the silicate, as mentioned previously, but when samples with their natural moisture in them were tested, they did not show much difference, using silicate and oxalate. See Fig. 14.

Hydrometer analyses were run using 50-g. samples and 20 ml. of silicate.

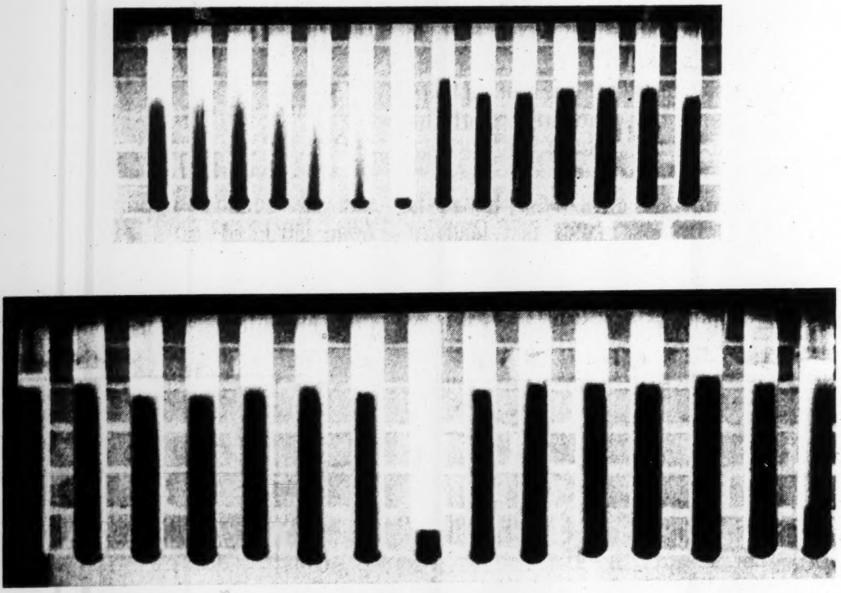

FIG. 14.—Soil No. 9. Pioneer Tests.

Top.—Air-dry samples at 22 hr.
Bottom.—Samples containing natural moisture at 20 hr.
Tubes at the left contain various amounts of silicate and those on the right various amounts of oxalate.

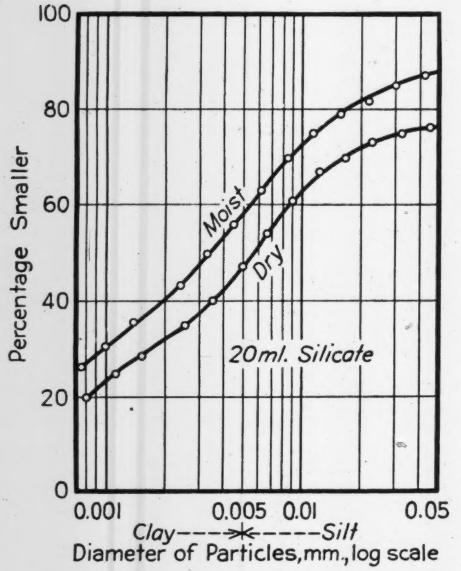

FIG. 15.—Soil No. 9. Comparison of an Air-Dried Sample with One That Had Its Original Moisture in It when Dispersed.

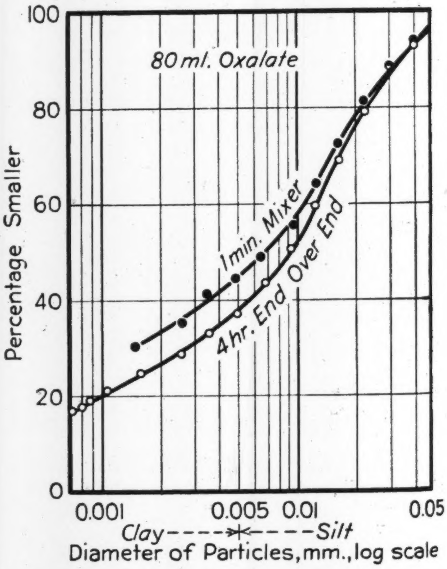

FIG. 16.—Soil No. 3. Comparison of Results Using 1 min. of Mixing with the Regular Mixer and 4 hr. of Mixing in a Glass Quart Jar in a Cylinder of the Deval Abrasion Machine.

Curves for these tests are shown in Fig. 15. The test with the moist sample gave 58 per cent clay and 30 per cent silt, while the air-dry sample showed 47 per cent clay and 30 per cent silt. It is interesting to note that the 20 ml. of silicate and the moist sample gave almost the same curve as the 80 ml. of oxalate and the air-dry soil.

Effect of Dispersion Machine:

The question has been raised by some concerning the adequacy of 1 min. of mixing with the standard mixer, while others have indicated that they felt the machine was too violent. Thinking of a substitute method, agitation was tried by placing quart glass jars in a cylinder of the standard abrasion machine for rock.[6] Samples of soil No. 3 were tested in this way, running the machine 1, 2, and 4 hr. Curves for the 4-hr. run in the Deval machine and for 1 min. with the standard mixer are shown in Fig. 16. One minute mixing with the standard machine gave larger percentages of the finer particles than did 4 hr. of the end-over-end method. Curves for 1 and 2 hr. of mixing were almost the same as the one for 4 hr., but they are slightly under it.

Some Base Exchange Considerations:

The very fine particles of soil are the ones that cause trouble in the sedimentation process. It has been shown that these fine particles of soil have on them what are known as adsorbed ions, such as Ca, Na, Mg, H, K, Ba, Al, Fe. Certain of these ions cause flocculation, while others do not. When the adsorbed ions cause flocculation, an attempt is made to replace them with one that does not. Baver reported in 1928[7] that potassium, sodium and in some cases magnesium have a tendency to deflocculate soils. In the deflocculation process a sodium salt is used, such as sodium silicate, sodium oxalate, sodium carbonate or sodium hydroxide.

The following represents what apparently happens when sodium silicate is added to a calcium clay:

$$\text{Ca-clay} + Na_2SiO_3 \rightleftharpoons 2\text{Na-clay} + CaSiO_3$$

Sodium-clay in water does not flocculate but the $CaSiO_3$ is sufficiently soluble in water to cause flocculation in spite of the fact the adsorbed ion is sodium.

If sodium oxalate is added to a calcium clay suspension, sodium-clay and calcium oxalate are formed:

$$\text{Ca-clay} + Na_2C_2O_4 \rightleftharpoons 2\text{Na-clay} + CaC_2O_4$$

The latter is very insoluble in water and has no effect on flocculation. If not enough of the agent is used, of course, not all of the calcium is replaced by the sodium, and if too much is supplied, the reaction reverses itself and calcium clay is again produced.

Another agent used to some extent is sodium hydroxide. Added to a calcium clay, we get:

$$\text{Ca-clay} + 2Na(OH) \rightleftharpoons 2\text{Na-clay} + Ca(OH)_2$$

The calcium hydroxide is quite soluble in water and flocculation takes place, even though the adsorbed ion is sodium.

Some soils contain calcium carbonate and since this is soluble in water, flocculation of the soil is inevitable. The only way to rid the soil of the calcium carbonate is to leach the soil with dilute hydrochloric acid, which will dissolve the calcium carbonate. At the same time, the H-ion will replace whatever other ion may be adsorbed on the soil. An acid soil is known to flocculate easily. Following the treatment with acid and washing with water, the acid soil may be treated with either sodium carbonate or sodium hydroxide to produce a sodium clay:

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[6] Standard Method of Test for Abrasion of Rock by Use of the Deval Machine (D 2 – 33), 1936 Book of A.S.T.M. Standards, Part II, p. 1040.

[7] L. D. Baver, "The Relation of Exchangeable Cations to the Physical Properties of Soils," *Journal*, Am. Soc. Agriculture, Vol. 20, No. 9, pp. 921–941 (1928).

$$2\text{H-clay} + Na_2CO_3 \rightleftharpoons 2\text{Na-clay} + HOH + CO_2$$

or

$$\text{H-clay} + Na(OH) \rightleftharpoons \text{Na-clay} + HOH$$

It is essential here also to use just the right amount of sodium carbonate or sodium hydroxide, as mentioned previously in connection with the calcium adsorbed ions.

Mention was made earlier that potassium or magnesium might be used as deflocculating agents. In considering the possibility of their use, some attention must be given to the products of the treatment. For example, using potassium hydroxide and a calcium clay, we get:

$$\text{Ca-clay} + 2KOH \rightleftharpoons 2\text{K-clay} + Ca(OH)_2$$

The $Ca(OH)_2$ is soluble and will cause flocculation. On the other hand, if the agent used were potassium oxalate, the products of the reaction would be potassium clay and calcium oxalate, and, since the latter is highly insoluble, flocculation would not occur.

It appears from the above that until there is some way of determining the adsorbed ions on the clay, as well as the presence of mineral compounds that will cause flocculation, the only thing that can be done is to try out various agents. Since there is no way of determining the amount of the replaceable ions on a soil, the exact amount of deflocculating agent must be found by a cut-and-try process.

Mixing Various Agents:

Several individuals indicated that they were combining two or more of the deflocculating agents in one test suspension, with the idea that if one did not work, another might. This idea was tried on a number of soils but with no success. The number of combinations of the variables becomes quite large and then a consideration of the possible base exchanges and resultant salts leads one to the conclusion that even though the right agent might be present to prevent flocculation, yet another agent might react to produce a salt causing flocculation.

CONCLUSIONS

While the number of tests involving the various variables was not large, there are a few conclusions that seem justified:

1. Sodium silicate is not the best deflocculating agent in the case of many soils, as has been reported previously.
2. The proper amount of deflocculating agent to use varies with different soils.
3. Certain soils contain a mineral or minerals that form a gel and when this gel is formed the sedimentation method of determining particle sizes is invalid. Decreasing the concentration of the suspension appears as a possible means of overcoming this effect. More work needs to be done, however, with the gel-forming soils.
4. For some soils at least the removal of the natural moisture content makes it more difficult to deflocculate the soil.
5. Use of several deflocculating agents in one suspension appears to be unwise.

Acknowledgments:

The major portion of this work was done by Donald L. Kirby, a senior in civil engineering, under the direct supervision of the author. Grateful acknowledgment for helpful suggestions is made to J. E. Lamar, Illinois Geological Survey; to John E. Gieseking, Associate in Soil Physics and Soil Survey, University of Illinois; and to Walter Worsham, Illinois Division of Highways.

THE GRADING-DENSITY RELATIONS OF GRANULAR MATERIALS

BY DONALD M. BURMISTER[1]

SYNOPSIS

The distribution of particle sizes and particle shape have a very important influence on the density of materials, and, therefore, special consideration is given to the grading of soils in order to bring certain physical factors into a more unified and consistent pattern. Density is not only influenced by the degree of fineness of a material and the range of particle sizes, but by the distribution of particle sizes, expressed in type grading curves. These factors, together with the effect of particle shape, are expressed quantitatively in terms of grading-density relations, which provide a means of estimating the influence of grading on density. These relations are important because density is an important factor in the supporting capacity of the natural soil and in the stability of slopes of granular soils.

Density is an important physical property of granular materials. It not only has an important bearing on the supporting capacity of the natural soil in place and on the settlement of foundations constructed thereon, but also has an important influence on the stability of the slopes of embankments and fills.

A study is made of certain physical factors which greatly influence the density of a given soil. The important factors determining density are:

1. The method of placing and the pressure used.
2. The distribution of particle sizes.
3. The shape of the particles.
4. The moisture content. For granular soils, this factor is not of so much importance as for fine-grained soils.

For a constant or standard method of placing, the influence of these physical factors is defined in terms of the grading-density relations.

In order to make the desired correlations and define these grading-density relations, the author has given special consideration to the definition of the grading or distribution of particle sizes of soils, in an attempt to describe the characteristic features of soils, and to furnish practical information of a more quantitative nature.

The grading analysis of soils is usually presented graphically in a grain size distribution curve with percentages of material finer by weight than any grain size plotted against the logarithm of the corresponding equivalent spherical particle diameter in millimeters. The grading analysis has, in the past, been evaluated in a more or less arbitrary fashion and certain terms have come into use which are of the nature of temporary adaptations.

A study[2] based on physical relations and on simplified statistical considerations shows that the size characteristics of soil may be represented by *three independent factors,* which bring many physical factors into a more consistent pattern:

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[1] Assistant Professor of Civil Engineering, Columbia University, New York City.

[2] D. M. Burmister, "A Study of the Physical Characteristics of Soils with Special Reference to Earth Structures," *Bulletin No. 6*, Civil Engineering Research Laboratories, Columbia University, June, 1938.

1. The degree of fineness of the soil, that is, the position of the grading curve on the scale of fineness.

2. The type of grading curve, that is, its shape, which indicates whether the soil has a normal distribution of grain sizes, or whether skewed toward the fine or coarse sizes, or of irregular distribution.

3. The range of particle sizes to take in the entire curve.

The most significant fact about the grading curve is that the area under the curve is a measure of the specific surface area of the material analyzed. The importance of surface area relations is recognized in certain chemical and metallurgical industries and in the cement industry. Practical use is made of the surface-area relations in the Gates' "crushing surface diagram"[3] in ore-crushing operations. Correlations show that the work performed in crushing a material is proportional to the increase in surface area, and that the area between the grain size distribution curves before and after crushing is a measure of the increase in surface area of the crushed material. It is well known that specific surface area plays a more and more important role in soil behavior as grain size decreases, for example, capillary phenomena, wetting characteristics, film character, physico-chemical surface reactions, etc.

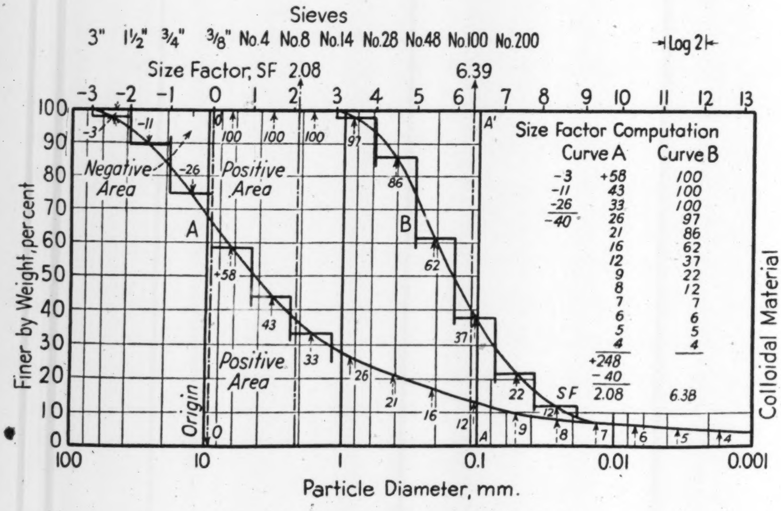

FIG. 1.—Analysis of Grading Curves.

Degree of Fineness:

An analysis based on surface-area relation of the grading curve, and involving and extending the ideas of Krey, Kozeny[4], and Abrams[5], shows that the degree of fineness of the soil may be represented by a mean grain size, defined by the length of the equivalent rectangular area, which is easily obtained by means of a planimeter. This mean is actually a weighted geometric mean and is also that grain size having the geometric mean surface area. This is the logical mean to use for a semi-logarithmic representation of the grading analysis.

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[3] A. F. Taggart, "The Handbook of Ore Dressing," pp. 495-496, John Wiley and Sons, New York City (1927).

[4] P. Forchheimer, "Hydraulik," pp. 56-63, B. G. Teubner, Leipzig (1930).

[5] D. A. Abrams, "Design of Concrete Mixtures," *Bulletin 1,* Structural Materials Laboratory, Lewis Institute, Chicago, Ill. (1918).

It is further convenient to express the degree of fineness in terms of a *size factor,* which is easily computed from the grading curve as given below and shown in Fig. 1. In order to obtain a significant area under the grading curve, an upper limit or *origin* must be defined. The origin of the logarithmic size factor designation is taken at the $\frac{3}{8}$-in. sieve (9.42 mm.) where there seems to be a definite change in the capillary tendencies and bulking effects of moist soils. The size factor scale of fineness is obtained by continuing the uniform scale of Tyler sieves at regular intervals of log 2 to a lower limit of 0.001 mm., where the material, for practical purposes, is of colloidal size. The size factor is made to increase as the material gets finer, because specific surface area of the material increases. Correlations[2] show that a definite size factor relation exists for the moisture or wetting requirements of granular soils and for the capillary tendencies.

The area under the grading curve is equal to the sum of the elementary areas, in which the ordinates (percentage y) are measured to the mid-point of the elementary area and the width of the area is log 2.

$$\begin{aligned} \text{Area} &= y_0 (\log d_0 - \log d_1) + \\ &\qquad y_1 (\log d_1 - \log d_2) + \cdots \\ &= (y_0 + y_1 + y_2 + \cdots) \log 2 \\ &= \Sigma\, y \log 2 \ldots\ldots\ldots\ldots\ldots (1) \end{aligned}$$

By equating this area to the area of the equivalent rectangle $OO'\,AA'$, a mean particle size is obtained:

$$\text{Area} = \Sigma y \log 2 = 100\,(9.42 - \log d_m)$$

$$\text{The mean grain size} - d_m = \frac{9.42}{2^{\Sigma y/100}}$$

$$= \frac{9.42}{2^{SF}} \ldots\ldots\ldots\ldots (2)$$

The size factor, thus defined, is a logarithmic designation of the degree of fineness of the soil and takes in the entire grading curve.

$$\text{Size factor, } SF = \Sigma \frac{y}{100} \ldots (3)$$

Equations 1 and 2 may be readily shown to be those of a particular kind of mean, namely, the weighted geometric mean grain size. In a sense the size factor is similar to the fineness modulus of Abrams[5], who took a summation for the "coarser than" grading curve, but considered only material coarser than the No. 100 sieve.

For coarse materials, the ordinates for the elementary areas are now laid out *above* the curve to the *left* of the origin at intervals of log 2, and the *percentages coarser are subtractive.* The size factor for very coarse materials may be minus, but this should not be confusing, when it is remembered that the area under the curve (or over) is a logarithmic function, The procedure is simply a method for obtaining a geometric mean. For materials with a minus size factor the capillary tendencies and bulking effects are negligible.

The significance of the size factor as a measure of surface area may be seen from a comparison of Eqs. 4*a* and 4*b*, which are similar in form:

Unit surface area

$$= \frac{\text{Total surface area}}{\text{Total volume of soil solids}}$$

$$= \Sigma \frac{N\pi d^2}{N\pi \frac{d^3}{6}} \times P \ldots\ldots\ldots\ldots (4a)$$

where N = the number of particles of size d, and
P = the percentage of that size.

Similarly—a log surface function

$$S = \frac{9.42 \times \text{constant}}{d_m}$$

$$= \text{constant} \times 2^{SF} \quad \ldots\ldots (4b)$$

$$= \frac{\text{Weight of soil}}{\text{sp. gr.}} \times \frac{6 \times 10}{9.42} \times 2^{SF}$$

$$= 240 \times 2^{SF} \text{ sq. cm. per 100 g. of dry soil}$$

$$= 1.18 \times 2^{SF} \text{ sq. ft. per lb.} \quad \ldots\ldots (4c)$$

Soils may be alike in one important respect, as far as the grading analysis can evaluate, in that they have the same degree of fineness and surface function. But soils may be unlike in other essential respects because of differences in the particular distribution of grain sizes. An important advantage of the size factor method is that the size factors of mixtures of soils may be easily obtained by simple proportion, because the size factor as a logarithm or exponent is additive (Eq. 1). The mean grain size may then be found from Eq. 2.

Type of Grading Curves:

It is an important and significant fact that the distribution of grain sizes has a marked influence on the density of a soil, whether placed loosely, compacted by some particular method, or as found in the natural state. A description of grading curves, as the six types shown in Fig. 2, is based on simplified statistical considerations and furnishes a basis for correctly interpreting the influence of the distribution of particle sizes on density.

The curves are drawn in this particular instance so that all have the same size factor; some of them, where possible, have the same range of particle sizes. As the range of sizes increases, the single-size type *A* material, which is represented by a nearly vertical grading curve, grades into one of the other types. The type *B* has an almost normal distribution of particle sizes with a typical S-shaped curve; the type *C* has equal percentages of all sizes and is approximately linear; the type *E* has a predominance of fine material with only a little coarse material and is skewed toward the fine sizes, while the type *F* has a predominance of coarse material with some fine material and is skewed toward the coarse size. The type *D* is the common type in glaciated regions and may vary from a mixture of some rock fragments in much fine material (D_E), to a mixture of considerable gravel or coarse sand in some fine material (D_F). Mixtures are always characterized by a hump in the grading curve.

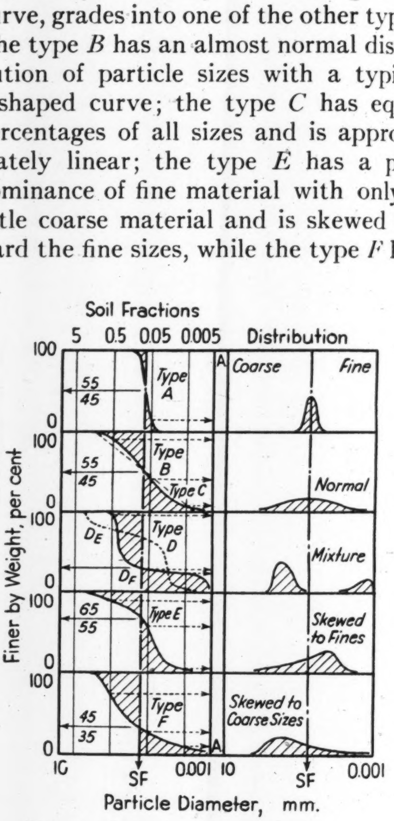
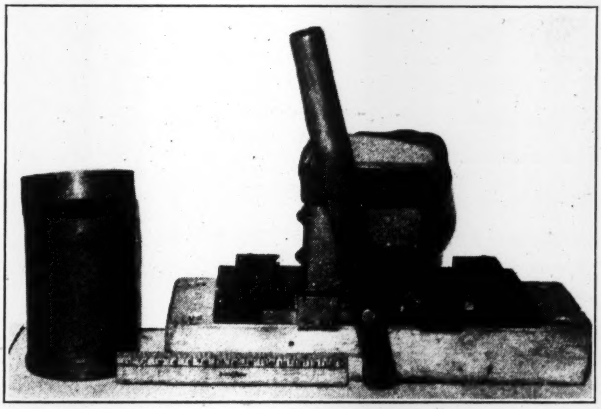


FIG. 2.—The Type Grading Curves.

A significant fact about these type grading curves is that the percentage corresponding to the size factor is not now an arbitrary constant, but varies in a characteristic fashion and in reality represents a *skew factor*. The percentage corresponding to the size factor or mean diameter may vary, depending on the amount of skew, from about 45 to 55 per cent for types *A*, *B*, and *C*, from about 55 to 65 per cent for types *E* and D_E, and from about 35 to 45 per cent for Types *F* and D_F. These type grading curves express relations which are quite distinct from any slope factors. Thus, for a given grain size or size factor, and range of particle sizes, a material is more definitely described by defining or specifying the type of grading curve and the amount of skew in terms of a percentage.

Range of Particle Sizes:

The size characteristics are finally defined in terms of the range of particle sizes. There is an important distinction between the shape of the grading curve and the range of particle sizes. The designation of range to take in the entire curve, since other factors describe the position and form, may be conveniently expressed simply as the ratio of the 98 per cent to the 2 per cent size with a lower limit of 0.001 mm., or as the number of size factor units between these extremes, with no reference to slope as such.



(*a*) A small cylinder. (*b*) Shearing-density apparatus.

FIG. 3.—Apparatus for Determining Density of Granular Materials.

The most important physical fact is that density is closely related to the distribution of particle sizes, as indicated in a general way in Fig. 2. The type *F* material is of special interest because it approaches the ideal grading curve for maximum density, as shown by the experiments of Fuller and Thompson[6], supplemented by those of Talbot and Richart.[7]

Grading-Density Relations for Dry-Granular Soils:

A study is now made of certain physical factors, which greatly influence the density of a given soil. The density will be between the limits of some loose condition and some very dense condition, whether placed artificially in an embankment by some mechanical means, or as found in the natural state. The *grading-density relations* for the limiting loose and dense conditions represent a simple and practical means of determining and expressing quantitatively the influence of such physical factors as distribution of particle sizes and of particle shape upon density.

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[6] W. B. Fuller and S. E. Thompson, "The Laws of Proportioning Concrete," *Transactions*, Am. Soc. Civil Engrs., Vol. LIX, p. 67 (1907).

[7] A. N. Talbot and F. E. Richart, "The Strength of Concrete; Its Relation to the Cement, Aggregates, and Water," *Bulletin 137*, Engineering Experiment Station, University of Illinois, October 15, 1923.

The loose condition is obtained by filling the container carefully with a large funnel in thin layers without permitting either compaction or segregation. The most effective method for obtaining the dense condition for granular materials is by means of vibration (rather than by rodding) such as obtained: (*a*) by giving a certain number of blows with the palm of the hand to the sides of the small container, (*b*) by using a vibrating concrete table for large containers, or (*c*) by using the shearing-density apparatus shown in Fig. 3, giving a slight back-and-forth vibrating motion to the handle until the material seems to stiffen up appreciably. The most dense condition is obtained by the latter method.

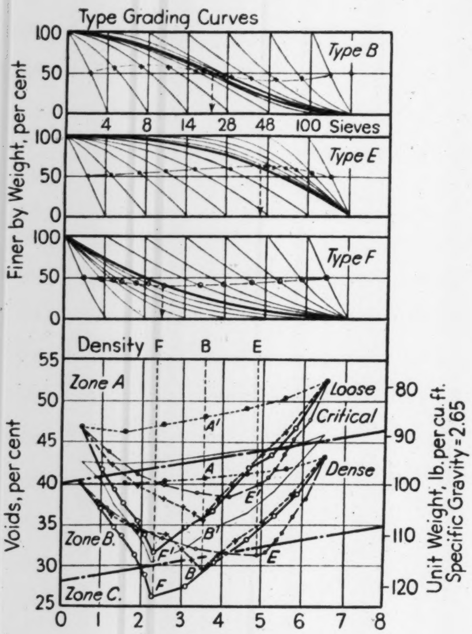

FIG. 4.—Grading-Density Relations.

The density is determined by weighing after turning down the rubber extension and screeding the material off level, knowing the tare weight and volume. The apparatus has a rubber diaphragm bottom to prevent loss of fines in the vibrating operation.

The percentage of voids is used as the measure of density rather than voids ratio, because the interpolated scale of unit weight is uniform. The relative position of the loose and dense conditions in Fig. 4 of each material now reveals distinctive differences in the character of the types, *B*, *E*, and *F* materials. The heavy grading curve in each case represents the whole material. Each of the other materials of the given type is obtained by sieving out, first, the coarser sizes one at a time, making the material of a narrower range and finer; and then by sieving out the finer sizes one at a time, making the material coarser. The size factor of each material is indicated on the grading curve and the density is plotted directly below on the vertical through this size factor. The influence of the distribution of particle sizes is now shown in Fig. 4. The relations in Fig. 5 disclose the important influence of particle shape on density.

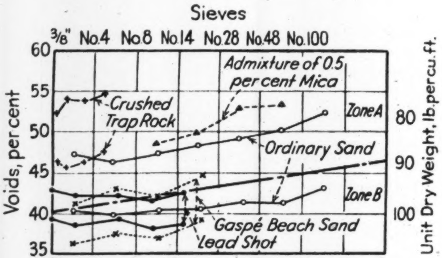

FIG. 5.—The Influence of Particle Shape on Density. Loose and dense conditions of the fractions.

The important facts brought out by these grading-density relations are summarized as follows:

1. *Degree of Fineness.*—For the same type of grading, range of particle sizes, and character of material (particle shape), the density tends to decrease as the size factor increases (particle size decreases). The trend of the zone lines gives the approximate trend of the decrease for narrow range materials, for example, the type *A* materials, or the materials of any other type having equal ranges of particle sizes. For wide range materials density becomes almost independent of fineness for dry granular soils.

2. *Type of Grading Curve.*—The type of grading curve has a most important effect on density. Other factors being equal, type *F* material is almost invariably the most dense. Type *B* is of intermediate density. Type *E* may or may not be more dense than the type *B*, depending on the character of the fine fraction. The single-size, type *A* materials are always the least dense.

3. *Range of Particle Sizes.*—The most important physical factor affecting density is the range of particle sizes. The most dense condition, other factors being equal, is obtained for that material having the greatest range of particle sizes, and the least dense by that having a very narrow range of particle sizes. The direction of the increase or decrease in range makes a considerable difference. The steepness of the curve for the predominantly coarse type *F* materials, for increasing range toward the fine sizes shows the greater importance of extension of range in this direction. For type *E* materials, which are predominantly fine, the extension of range toward the course sizes has a more marked effect on density. Type *B* materials are affected about equally in either direction.

4. There appears to be a consistent and characteristic relation or spread between the loose and dense conditions for each soil and for any given method of placing, which amounts to a change of about 6 per cent at the coarser sizes to about 10 per cent at the finer sizes in the percentage of voids, and 10 to 15 lb. in the density.

5. *Particle Shape.*—The effect of subangularity, and to a greater extent angularity (crushed rock and rock splinters), is to decrease density very materially in both the loose and dense conditions. The addition or presence of detrimental soil constituents such as small quantities of micaceous material (even 0.5 per cent) is materially to decrease density. The spherical shot is used as a norm. In contrast to granular soils, the density of shot is greater and appears to be independent of particle size and the spread is smaller. The fact that the soils show a tendency for a decrease in density with decrease in particle size indicates the subangular character of all natural soils. It is somewhat surprising that materials of smooth, flat, and elongated particles, such as the Gaspé beach sand, produce a greater density than shot in the dense condition, and considerably greater than ordinary sand. This may be due to the tendency for the particles to orient themselves flatwise and nest closely. This may explain in part the excellent character of certain earth roads made with such materials as the coarse fraction.

Studies[2] have shown that these grading-density relations carry all the way through to the very finest of materials and tend to bring into a more consistent and unified pattern the data on soil compaction and stabilization.

Between the limiting loose and dense conditions, but somewhat nearer the dense state, is a significant physical property of cohesionless, granular soils—the critical density[8], which Casagrande has defined as that volume or density of a soil at which continuous shearing deformation can take place at constant volume with a constant shearing stress. This is the plastic state of granular materials. This critical density has been investigated by Kayganovich[9] by

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[8] Arthur Casagrande, "Characteristics of Cohesionless Soils Affecting the Stability of Slopes and Earth Fills," *Publication No. 173*, Graduate School of Engineering, Harvard University (1935-1936).

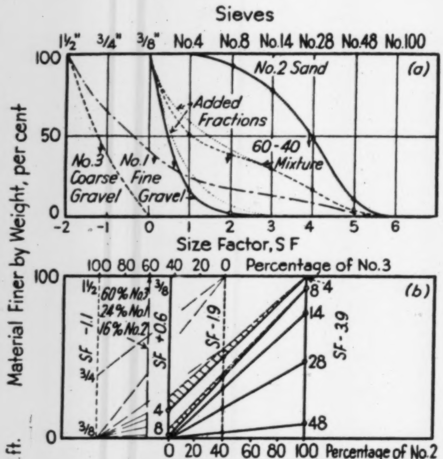

FIG. 6.—The Size Characteristics and Grading-Density Relations of Mixtures of Granular Materials.

means of the simple shearing-density apparatus illustrated in Fig. 3. The critical density is obtained by giving the lever the full throw back and forth 25 times, the cross-sectional area of the soil mass being alternately deformed from a square to a diamond shape in the process. The density is determined by weighing after screeding the material off level, knowing the tare weight and the volume.

The relation of the critical density to the loose and dense states depends on the type of grading curve and the range of particle sizes and indicates the decrease or expansion in volume of the soil mass required to reduce it to the critical density. These relations are shown in Fig. 4 for types *A* and *F* materials only.

A practical application of these ideas is made in a study of the difference in the characteristics of materials obtained by combining two materials shown in Fig. 6(*a*). Material No. 1 is a coarse narrow range type *AF* material and material No. 2 is a finer, wider range type *B* material. The grading curves are transposed to the diagram of Fig. 6(*b*). using the size factor method of analysis. The percentages corresponding, in this case, to the sieve sizes are projected onto a vertical line, as *A*-*A* in Fig. 2. The soil fractions chosen will vary according to the purpose of the study (Tyler sieves, log 2 intervals or any other interval as desired). The grouping of these percentages is also characteristic and serves to indicate both the type of grading curve and the range of particle sizes. These soil fractions are now plotted on a vertical line through their respective size factors. By applying the additive principle of the size factor method, straight lines joining the respective fractions of each material in Fig. 6(*b*) now represent every possible combination of these two soils—the size factor and the percentages of the fractions being directly proportional to the proportions of the two soils used in the combination.

The addition of a small amount of material No. 2 greatly increases the range, but the grading curve is not regular as shown by the grouping or crowding together of soil fractions for the 60 per cent of No. 1 and 40 per cent of No. 2 mixture. If the fractions for this mixture are transposed back to the grading curve of Fig. 6(*a*), which is easily done by means of dividers, it is seen that the curve is a typical *D* type with a hump in it, corresponding to the point where the fractions are crowded together in Fig. 6(*b*). A study of the grouping of the fractions for all combinations in Fig. 6(*b*) shows that the type *F* grading curve of maximum density cannot be obtained by any mixture of these two particular soils. The investigations of Talbot and Richart[7] have shown that the density is greater for smooth regular grading curves of the Talbot grading, that is the type *F* curve, and is less for irregular grading curves. This method provides a simple means of studying the characteristics of mixtures, of estimating the best proportions, or of the need of adding a third material to produce a better proportion, such as indicated by the dotted lines in Fig. 6(*a*) and (*b*). which smooth out the deficiency of sizes and produce a more regular grading curve with each size bedded in approximately the proper proportion of the next size smaller—the Type *F* grading curve.

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[9] John Kayganovich, "Investigation of the Density Characteristics of Granular Materials," Thesis submitted to the Department of Civil Engineering, Columbia University (1938).

The influence of the properties of the mixture on the density is now clearly shown by the grading-density relations in Fig. 6(*c*). The most dense mixture is obtained by the 60 per cent of No. 1 and 40 per cent of No. 2 combinations—a typical *D* type material. The addition of the proportions of the materials indicated to smooth out the deficiency of these sizes, and produce a curve most nearly of type *F* results in an appreciable increase in density as noted. This represents the maximum density that can be obtained from these materials, although the curve is still a type *D*. Further addition of material to smooth out the curve changes the type away from the type *F* and actually results in a decrease in density. The only way to obtain greater density is to sieve out material to smooth the hump or better to increase or extend the range of particle sizes in either the course or fine direction by the addition of a third coarser or finer material.

The marked effect of increasing the range in the coarse direction, for example, is shown by the right-hand branches of the density curve in Fig. 4 for the type grading curve. An almost linear relationship exists between density and increasing range. Equal increments of range in size factor units produce approximately equal increments of density, the size of the density increment varying markedly, however, with the type of grading curve. By extending the right-hand branches of the density curves, an estimate of the increase in density can be obtained from Fig. 4 for materials having a greater range of particle sizes, which approximate the type curves shown, for example, the addition of material No. 3 of coarser sizes.

Thus it is possible to study the size characteristics and grading-density relations of mixtures and proportion them to meet the requirements of maximum density, or estimate the density of such mixtures. The grading-density relations of granular materials describe basic physical relations and give quantitative expression to the important influence of the distribution of particle sizes and of particle shape upon density. These relations are of fundamental importance because density is one determining factor in the supporting capacity of the natural soil, and in the stability of slope and earth fills of granular materials. The properties of granular soils in the dry and inundated condition are practically identical, and they are affected only to a slight extent by variations in moisture content in the partially saturated condition. The bulking effect in fills is negligible in coarse materials and becomes more important for the finer materials.

The most significant facts are that each material can exist in a certain loose condition in its natural state or in some artificial state in embankments, and can be compacted only to a certain dense state by external loadings, vibrations, rolling, shrinkage, etc., with a large or small spread between the loose and dense conditions. Type *F* material in its loosest state may have a greater unit weight than type *B* or *E* materials in their densest state, but considerable volume change, shrinkage or settlement can take place. The most important thing is the relation of the actual density of the natural soil or of soil in embankments to its limiting loose and dense condition, and particularly to the critical density.

If the natural soil has a density greater than the critical, it will have a relatively higher supporting capacity, and if placed in an embankment at a density greater than the critical, it will be more stable and the shrinkage will be considerably smaller. These facts will be of significance in relation to its behavior under the given circumstances. Thus, it is possible, through a study of the grading-density relations of granular material to determine basic physical properties of the material, which have an important bearing on the supporting capacity of the natural soil and upon the stability of slopes in embankments.

DISCUSSION

Mr. I. F. Morrison[1] *(presented in written form)*.—In the mechanics of granular masses, we like to believe that the mechanical phenomena exhibited by masses of relatively coarse particles have their qualitative counter-parts when masses of fine particles are considered. We have, of course, no assurance that this is so, but nevertheless find it expedient to make such an assumption. On the other hand, with very fine particles physical phenomena, present but insignificant in the case of coarse particles, become the predominating influence, thus obscuring and overpowering the mechanical behavior of the fine particles which make up the mass. Still, there is much to be learned from direct observation of masses of relatively coarse particles and Mr. Burmister's paper (the experimental part of which deals with sands lying above the No. 100 sieve) forms an excellent example of what can be done as well as a valuable contribution to the science of density of granular materials.

There can be no doubt of the outstanding importance of density in its bearing on earthwork engineering, and the idea of the density-state and its possible variations, produced both artificially and by natural causes, with their consequent effects, is rapidly coming to be recognized in engineering practice. Rigidity of a mass, however, often called stability, is not solely a matter of density or closeness of packing. Studies of the space-lattice structures of crystals show that closeness of packing of the atoms is not indicative of the qualities called hardness or rigidity and metallurgists have for some time been striving for clarification of ideas along the lines of a distorted or interrupted lattice related to the concept of interlocking.

Interlocking is more readily visualized in granular particles than in atoms, and that it plays an important part cannot be doubted, even to the extent of becoming more important in certain cases than density. This phenomenon depends primarily on particle shape and there appears to be a natural tendency for particles to become interlocked, although vibration produces an orientation and consequently an unlocking effect which may, while increasing the density, lead to a decrease in rigidity. As an adjunct to the phenomenon of rigidity due to particle interlocking, inter-particle friction is of considerable importance in the case of dry materials and therefore the character of the particle surface also plays a part. The author might well have added this item in his list of important factors.

In order to make these remarks clear, the writer presents some extreme cases involving materials having type A grading curves. The accompanying Figs. 1 and 2 show photographs of a mass of small nails and of Ottawa standard sand, respectively. A comparison of these small piles shows that the mass of nails stood with a vertical face while the sand flowed out to a comparatively flat slope. These piles were produced by placing the respective materials in boxes of the same size. In each case the boxes were vibrated to obtain a firm packing and they were then carefully inverted onto a horizontal surface. In the case of the pile of nails, which possessed considerable rigidity, the volume of voids was 77 per cent, and in the case of the sand, which had very little rigidity, the volume of voids was 32 per cent.

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[1] Professor of Applied Mechanics, University of Alberta, Edmonton, Alta., Canada.

In order to obtain a more dense packing, in the case of the nails, they were carefully packed into a rectangular pile all lying parallel to one another. When released, the pile flowed to a slope of 22 deg. to the horizontal, and the voids, before flowing, were 30 per cent. The angle of repose for the sand, shown in Fig. 2, was found to be 20.5 deg.

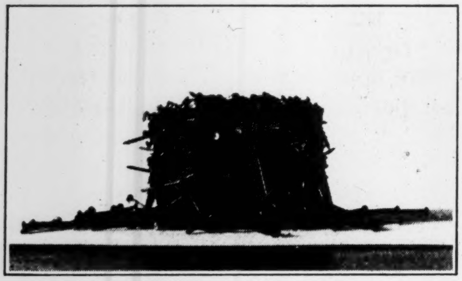

FIG. 1.—Mass of Small Nails.

These simple cases show that greatest rigidity is not necessarily associated with greatest density and that particle shape, due to the phenomenon of interlocking, may be of prime importance in the attribute called rigidity. The writer has seen piles of dry coal standing at a steep angle. In this case there was a wide range of particle size but many of the finer fragments had splinter-like shapes, which would account for the large angle of repose.

Particle shape and arrangement are both much more difficult to evaluate than particle grading, but some further studies on particle shape - density relations would form an important addition to the science of granular materials. Possibly Mr. Burmister can work out in the future some relationships between these factors and the rigidity of a granular mass upon which so much depends.

MR. DONALD M. BURMISTER[2] (*author's closure by letter*). —Mr. Morrison has made some very interesting comments, particularly with respect to the rigidity and stability of granular masses, which have an important bearing on earthwork engineering. He has pointed out that rigidity or stability is not only a matter of density or closeness of packing but is due also to interlocking of grains. Interlocking is a matter both of particle shape and of coarseness.

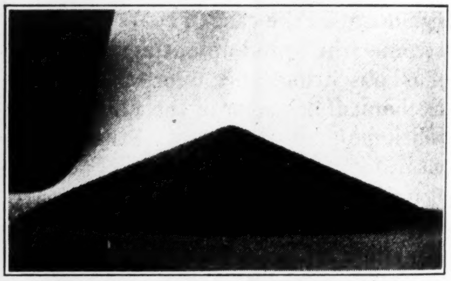

FIG. 2.—Mass of Ottawa Standard Sand

In order to get a clear picture of the shearing phenomena in granular materials, one must consider not only the usual shearing stress-strain relations but also the shearing stress-strain-volume relations. The volume changes that accompany shearing deformations are of fundamental importance. These volume relations are expressed in terms of the *Critical Density*[3] which is defined as that particular volume or density-state at which continuous shearing deformations can take place without volume change and at constant stress. This represents, for granular materials, the equivalent of the plastic state—a change in shape of the soil mass without change in volume. The shearing stress-strain-volume relations are then manifested by the fact that loose soils contract in volume to the critical density during shear while dense soils must expand in volume to the critical density during shear.

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[2] Assistant Professor of Civil Engineering, Columbia University, New York City.

[3] Arthur Casagrande, "Characteristics of Cohesionless Soils Affecting the Stability of Slopes and Earth Fills," *Publication No. 173*, Graduate School of Engineering, Harvard University (1935-1936).

A particular instance of the critical density is the dry rock flow or rock glacier which is of quite frequent occurrence where the talus from a cliff accumulates in sufficient quantities to keep the movement in progress. What the peculiar set of circumstances is that sets the mass of large and small angular fragments or masses of rock in motion and keeps them in continuous motion on a very flat slope of about 5 deg. is not known, but the fact remains. In order for the mass to move, it must be at the critical density, at least in the zone of greatest movement, and all interlocking effect is destroyed by the motion or has never been allowed to build up. The angle of repose of the rock flow is flatter than 5 deg. Yet the same material under different conditions would form a stable slope with an angle greater than 45 deg. as noted in dry piles of coal, concrete aggregates, etc., possibly in a much less dense state, largely because of the interlocking effect. Slides in such piles of materials are usually purely a surface phenomena and do not involve mass movements.

But it must be remembered that the slope or angle of repose in practically every case is purely a surface phenomenon and does not represent the angle of friction. The angle of friction is a function of the strain-volume relations; for example, it may be as high as 47 deg. in a *dense*, coarse-grained material and may be as low as 30 to 35 deg for the same material in the loose state. This very considerable difference comes about from the fact that for densities greater than the critical, an expansion in volume must occur in order to permit shearing distortion to take place within the mass: that is, rolling, sliding of particles over one another, or relative displacement—whether there is interlocking or not. If the expansion is prevented or restricted by the weight of the overlying mass of material, or can only take place with difficulty, then the mass will be very stable. On the other hand, in loose masses, shearing distortions permit a volume decrease to accompany them and during motion the angle of internal friction can never exceed the value at the critical density. If there is interlocking of angular materials for densities above the critical, these masses will be stable as long and only as long as shearing deformations in the mass do not grow to the point where the interlocking effect begins to break down and a volume decrease must accompany the shearing distortion. This situation is like the king log in a log jam. Kick out this one log and the whole mass becomes mobile and flows out suddenly, sometimes before the loggers can all escape to safety. Therefore such conditions,

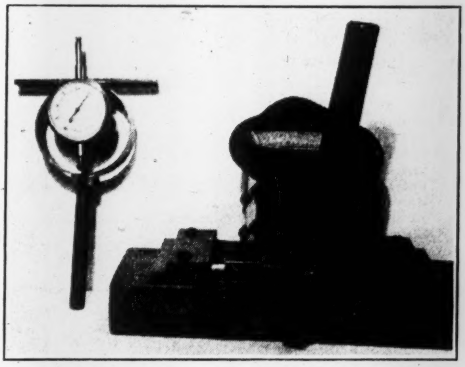

FIG. 3.—The Shearing-Density Apparatus.

Showing the penetration device consisting of a calibrated loading ring with a $^1/_2$-in. needle attached.

apparently very stable, may become suddenly unstable, if vibrations kick loose the keystone of the rock arch system within the mass, and the apparently steep stable surface slope is flattened by internal mass movements to a new stable condition.

In order to get a clearer picture of the stability conditions of granular masses as affected by grading, particle shape, density, etc., the following tests were made. In order to obtain some indication of internal stability—that is, resistance to displacement—a simple penetration test was made by the apparatus illustrated in the accompanying Fig. 3, which consists of a $\frac{1}{2}$-in. needle fitted to a calibrated loading ring. The results are given in the accompanying Table I. The resistance to penetration has long been considered to be a measure of stability and supporting capacity and, in this small scale test, illustrates the very considerable differences in penetration resistance for different materials. The penetration resistance was determined in each case for the limiting density-states, loose and dense, and for the critical state as produced by the shearing-density apparatus. The tests were made in the apparatus itself and therefore the results are comparable among themselves.

TABLE I.—RESISTANCE TO PENETRATION IN KILOGRAMS OF A $\frac{1}{2}$-IN. NEEDLE FOR A PENETRATION OF 2 IN.

Type of Material	Density-States		
	Loose	Critical	Dense
1. Ordinary sand and fine gravel (sub-angular):			
Type F grading	1.5	3.2	16.2
Type E grading	1.5	3.2	18.8
Type B grading	1.9	3.6	16.8
2. Ottawa sand No. 20–No. 30 mesh. Well-rounded grains	0.4	1.4	4.6
3. Ordinary sand No. 14–No. 28 mesh	0.4	3.1	4.5
4. Gaspé Beach sand—smooth rounded flat and elongated grains			
Sieves ⅜ in.–No. 4	4.2	12.5	55.7
No. 4–No. 8	1.2	4.8	9.0
No. 8–No. 14	1.1	3.5	8.5
No. 14–No. 28	0.4	3.0	10.0
5. Trap rock—very angular sharp fragments, mostly long splinters			
0.187 to 0.131 in	2.0	47.0	59.3
0.098 to 0.065 in	2.8	12.2	55.2

A study of this table brings out a number of interesting facts. Consider first the effect of particle shape:

1. The rounded Ottawa sand grains offer the least resistance in all density-states. Ordinary sand of about the same separation size has about the same resistance.

2. The smooth rounded but flat and elongated particles of the Gaspé Beach sand smaller than the No. 4 sieve likewise have rather low resistances.

3. But for materials coarser than the No. 4 sieve, the resistance markedly increases due largely to the difficulty of displacement, partly due to the effect of the small size of the apparatus.

4. The trap rock, due to interlocking even in the small sizes, has an extremely high resistance to penetration.

5. *Conclusion.*—Narrow range *fine* materials have low resistance to penetration in all density states. In contrast, narrow range *coarse* materials have a high resistance, due to particle interlocking and greater resistance to relative displacement and volume expansion. The results of the tests on the coarse materials may be more affected by the restraint offered by the box itself. All range materials show a considerable increase in penetration resistance. For the same relative density and grading, angular materials show a much higher resistance to penetration.

Now consider the effect of density itself. For all materials the resistance to penetration, regardless of particle shape, is very low. In most cases, there is very little increase in resistance to penetration even at the critical state which represents a relative density of about 50 to 60 per cent, because displacement can occur readily in this density-state, with the exception of the coarse sizes and the trap rock. In every case, there is a marked increase in the resistance to penetration in the dense state because displacement must cause a considerable volume increase, which is restricted. The range in which cohesionless materials may be unstable is a rather broad zone extending from this upper critical density, as determined by the shearing-density apparatus to a relative density of about 60 per cent. For such cohesionless materials to be stable under a tendency for shearing action, the relative density—$\left(\frac{e_{\text{loose}} - e_{\text{natural}}}{e_{\text{loose}} - e_{\text{dense}}}\right) 100$ — should be greater than 70 to 80 per cent.

The important conclusion is that a penetration test has great possibilities for determining the relative density and stability of natural granular masses, for material of not too coarse grain sizes, if studies on a larger scale could establish the fact that a fairly simple and definite relation existed between resistance to penetration, relative density, and stability, for different types of gradings and particle shapes.

A METHOD FOR THE EMBRITTLEMENT TESTING OF BOILER WATERS[1]

By Frederick G. Straub[2] and T. A. Bradbury[2]

Synopsis

A method for testing samples of boiler water whereby embrittlement is produced in a sample of steel has been devised. This method of testing simulates boiler operation and eliminates several assumptions heretofore made. As a result of tests made on a large number of boiler water samples from boilers operating between 150 and 1400 lb. pressure, conclusions have been reached relative to water treatment for the prevention of embrittlement. It has been found that the presence of sodium chloride aids sodium sulfate in preventing embrittlement at pressures up to 350 lb. At steam pressures above 350 lb. sodium sulfate has practically no inhibiting action, but small amounts of aluminate are very effective in this range. Utilization of these results is being made in several power plants.

The problem of the cause of embrittlement in steam boilers and methods of its prevention has been very thoroughly studied since the early days of the occurrence of this insidious type of steam boiler distress. Much research work has been conducted in order to answer the various questions which have been asked in regard to this type of boiler failure and these researches have been quite fruitful in their results. However, the boiler operator is still faced with the problem of trying to determine whether his particular boiler water is embrittling in nature and if it is, what he has to do to make it non-embrittling.

It was with the purpose of answering these questions for the boiler operator that the work herein described was started. Early in the work, it was realized that there was need of a simple method of testing, whereby a sample of boiler water could be tested in such a manner as actually to produce embrittlement in a piece of boiler steel under conditions directly comparable to boiler operation. Embrittlement has been reproduced in boiler steel but under conditions which made various assumptions necessary. First, the concentrations of the chemical involved were about one thousand times that existing in the boiler; second, the steel was tested in a container so that a large volume of concentrated solution surrounded the test specimens; and third, actual boiler waters could not be directly tested under such conditions.

A survey of the instances of embrittlement found in steam boilers showed that they always occurred in an area where a capillary space existed and where concentrated stresses might occur. Leaks to the outside were apparently not necessary. Experimental work conducted by one of the authors[3] showed that concentrated solutions of sodium chloride could be produced in capillary spaces in the absence of leaks. However, this method had limitations in that if sodium hydroxide were to be concentrated under similar conditions the increase in boiling point might limit the ultimate concentrations reached. Examination of boilers in which embrittlement has taken place left no doubt but that concentrated solutions of sodium hydroxide have existed in these areas.

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[1] The results of the research report are part of the investigation conducted under a cooperative agreement between the Utilities Research Commission, Inc., of Chicago, Ill., and the Engineering Experiment Station, University of Illinois, Urbana, Ill. These results are released by permission of both parties of the cooperative agreement.

[2] Research Associate Professor, Chemical Engineering, and Laboratory Assistant, respectively, University of Illinois, Urbana, Ill.

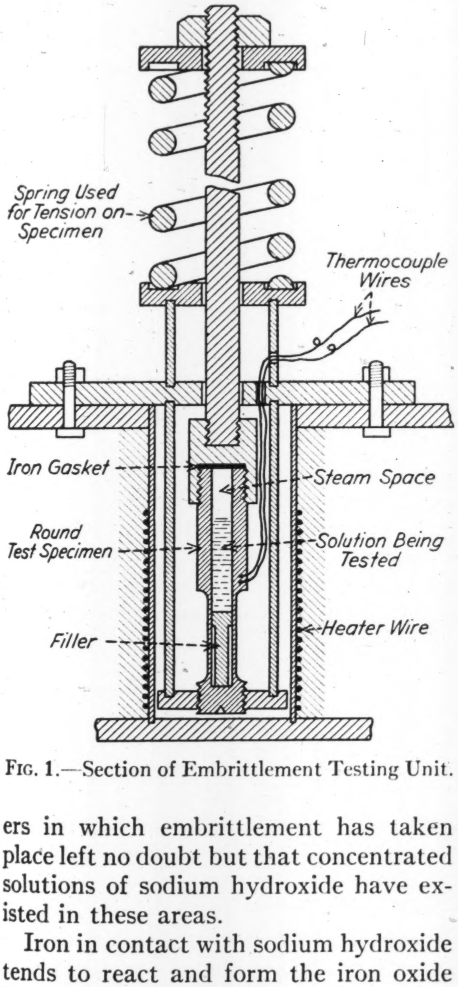

FIG. 1.—Section of Embrittlement Testing Unit.

Iron in contact with sodium hydroxide tends to react and form the iron oxide and liberate hydrogen at relatively low concentrations of sodium hydroxide and at boiler water temperatures. If this reaction takes place, it is possible for the primary concentration to take place in a manner similar to the sodium chloride tests reported. If this concentration were to take place in a boiler seam where a small volume of water is in contact with a relatively large area of iron the reaction of a partially concentrated sodium hydroxide solution with the iron to evolve hydrogen would remove the water and concentrate the caustic, thus causing the reaction to proceed further. The following reaction might be the one involved:

$$3Fe + NaOH + 4H_2O \rightarrow Fe_3O_4 + NaOH + 4\ H_2.$$

It will be noticed that the sodium hydroxide is not changed during the reaction but merely becomes more concentrated as the reaction proceeds. In this manner a very concentrated caustic solution could be produced without the temperature becoming high enough to boil the water out of the solution. The testing of a concentrated solution surrounding a test specimen where free circulation is possible precludes the possibility of this second reaction taking place. Furthermore, the concentration within a seam would produce other conditions which would not be reproducible under the older methods of testing.

To take advantage of these ideas, the method of testing should involve the placing of a capillary space adjacent to the steel under tension. The water to be tested should have access to this capillary space, and free circulation must be prevented. Figure 1 shows a section of a test unit devised to test dilute or concentrated solutions. The test specimen is made of a known steel. In these tests, S.A.E. No. 1020 hot-rolled steel has been used, although any type of steel might be used. The steel specimen is 1 in. round and about 5 in. long. A hole 0.500 in. in diameter is bored into the specimen and a portion of the steel removed near the bottom so as to give a reduced section. When this specimen without the filler was partially filled with a solution of caustic soda containing silica, assembled in the test unit, heated, and stressed, by compressing the spring, it was found necessary to have concentrated solutions of caustic present before failure resulted. This was as expected, since this method of testing is similar to the older ones used except that the solution is inside the specimen and thus eliminates packing glands along with potential points of leakage. In this test unit, a sample of a solution made by adding 250 g. sodium hydroxide to a liter of water plus 14 g. of SiO_2 (added as sodium meta silicate—$Na_2O \cdot SiO_2 \cdot 5H_2O$—a very pure crystalline product was furnished by Philadelphia Quartz Co.) caused cracking, while the same solution diluted with water so that the sodium hydroxide became 50 g. per liter, did not produce failure. The load applied in these tests was 50,000 lb. per sq. in. (applied at the test temperature) and the tests were run at 500 F.

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[3] "Embrittlement in Steam Boilers," *Bulletin 216*, University of Illinois, Engineering Experiment Station.

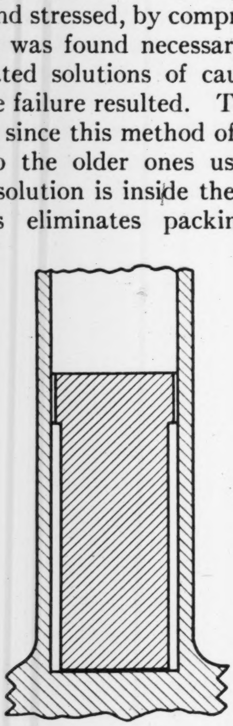

FIG. 2.—Section of Specimen and Filler.

The method was then modified[4] by inserting the small solid steel filler inside the test specimen as shown in Fig. 2. The filler is made a few thousands of an inch smaller than the inside of the specimen at the top and reduced still smaller at the bottom. This allows a capillary space to exist in contact with the stressed area of the specimen. This design of filler was used so as to allow the test solution to have access to the capillary space where it could concentrate, in contact with the stressed steel, without removing steam from the test specimen. This is very similar to the stressed areas in the boilers where embrittlement takes place. When dilute solutions of caustic soda were tested in this unit, failure resulted.

TABLE I—EFFECT OF TEMPERATURE AND TYPE OF LOADING ON HOLLOW SPECIMEN WITH FILLER.

Solution used contained 800 p.p.m. NaOH 80 p.p.m. SiO_2

Test	Temperature, deg. Fahr.	Load Applied	Load Applied, lb. per sq. in.	Results of Tests	
				Break	No Break
No. 1710.....	300	Hot	40 000	2 hr.	
No. 1610.....	400	Cold	45 000	....	25 days
No. 1617.....	400	Cold	50 000	2 days	
No. 1619.....	400	Cold	55 000	10 days	
No. 1608.....	400	Cold	40 000	6 days no break; load hot 45 000, break 3 hr.	
No. 1612.....	400	Hot	40 000	2 hr.	
No. 1618.....	400	Hot	40 000	2 hr.	
No. 1648.....	400	Hot	40 000	2 hr.	
No. 1686.....	400	Hot	35 000	5 hr.	
No. 1689.....	400	Hot	30 000	19 hr.	
No. 1603.....	500	Cold	40 000	2 to 4 hr.	
No. 1604.....	500	Cold	35 000	4 to 15 hr.	
No. 1605.....	500	Cold	30 000	6 days no break; load hot 35 000, break 1 hr.	
No. 1606.....	500	Cold	35 000	0 to 10 hr.	
No. 1685.....	500	Hot	30 000	breaks	
No. 1687.....	600	Hot	35 000	0 to 10 hr.	
No. 1688.....	600	Hot	30 000	$1\frac{1}{2}$ hr.	
No. 1692.....	600	Hot	25 000	....	10 days

Table I gives the results of tests run at temperatures between 300 and 600 F.

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[4] Patent applied for.

using a solution containing 800 p.p.m. of NaOH and 80 p.p.m. of SiO_2. The solution used in the hollow specimen tests without the filler, in which no failure resulted, was approximately 50,000 p.p.m. or 60 times the concentration of the dilute solution which caused failure when the filler was used. As shown in Table I, two methods of loading were used, one where the load was applied at room temperature and the other where the specimen was heated to the test temperature, held there for four or five hours, and then subjected to the desired stress. These results show that apparently the insertion of the filler has brought about a condition whereby dilute solutions will concentrate in the capillary space and in the presence of sufficient stress produce failure of the steel. In test No. 1692 where no failure resulted, the solution above the filler at the end of the test had only 60 p.p.m. of sodium hydroxide in solution. This showed a reduction from 800 p.p.m. to 60 p.p.m. The sodium hydroxide had

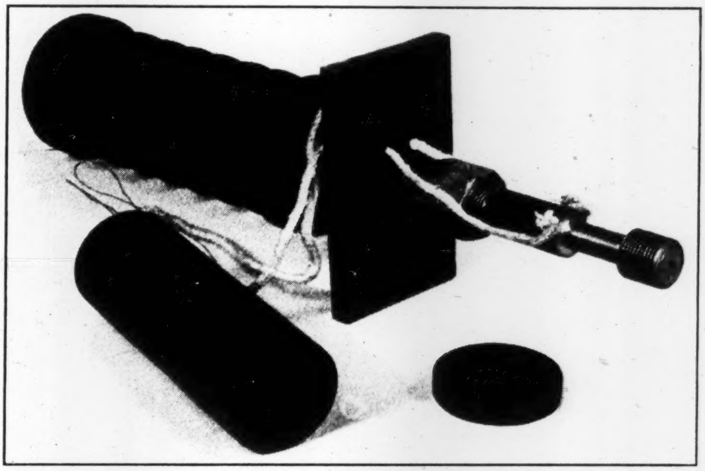

Fig. 3.—Specimen Screwed onto Top Holder.

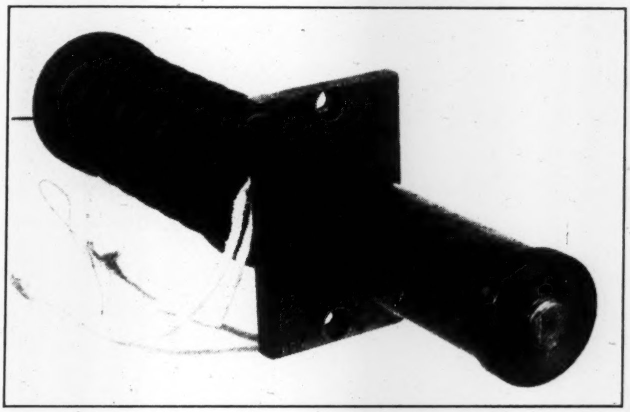

Fig. 4.—Test Unit Completely Assembled.

been concentrated in the capillary space with the reduction of concentration in the solution above. In this test the upper volume of solution was around 6.5 ml. with a maximum volume of 0.5 ml. in the capillary space. The removal of 740 p.p.m. of NaOH from the top solution and concentrating in the lower capillary would increase this concentration to 10,000 p.p.m. as a minimum value. If part of the capillary were filled with H_2 gas or steam, this concentration would be much higher. Examination of the lower part of the specimen at the end of the test gave definite evidence of the presence of concentrated solution by the surface attack on the steel.

Subsequent tests have shown that failure has resulted with the use of solutions containing as low as 100 p.p.m. of sodium hydroxide. This indicates that this method of testing should be very suitable to test boiler waters to determine their embrittling characteristics. Tests run on boiler waters in this unit should give data relative to which salts are causing or preventing the embrittlement. It should be quite suitable for such testing since no assumptions are made and the boiler water sample contains all the chemicals present in the particular boiler. If failure results, it would be logical to state that there is a potential possibility of failure taking place in the boiler, while, if no failure results under these conditions, the boilers should operate free from embrittlement.

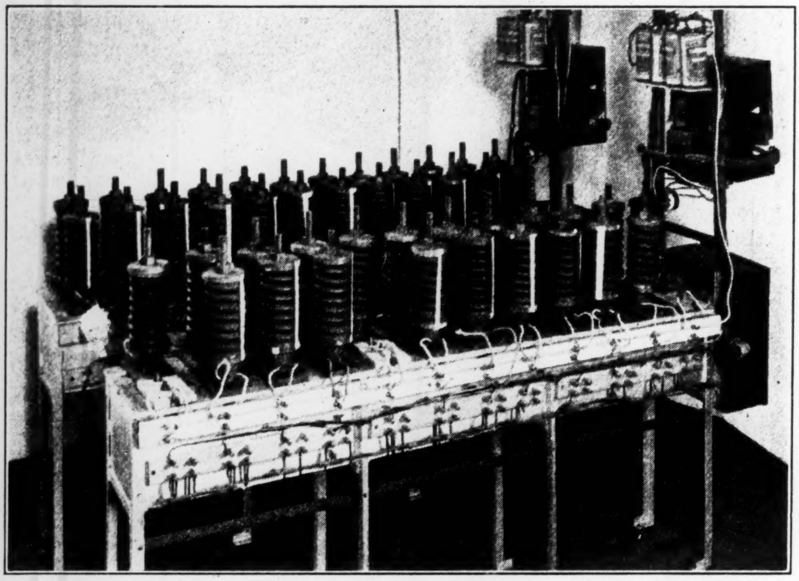

FIG. 5.—Test Units, Heating Furnaces and Temperature Controllers.

In order to check this method of testing, boiler water samples were collected from power plants where embrittlement was being experienced as well as from plants not having any trouble. More than 200 such boiler waters[5] have been tested at temperatures between 360 and 600 F. (150 to 1500 lb. steam pressure).

Figures 2, 3 and 4 show views of the

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[5] These waters were analyzed as follows: Total alkalinity, chloride and sulfate (gravimetric) by A.P.H.A. method. Silica and R_2O_3 gravimetrically, using perchloric acid for the silica.

testing unit and illustrate how it is assembled. Figure 5 shows several batteries of furnaces, testing units and temperature controllers, capable of testing 48 waters at one time. The procedure used consisted of placing the sample of water (7 ml.) in a new specimen, inserting the oxidized filler, sealing the specimen into the holder, peening the thermocouples (iron and constantan) into the side of the specimen and assembling the test unit (Fig. 4). The assembled unit was then placed in a hot furnace where it was held at the desired test temperature (±15 deg. Fahr.) for a period of four or five hours. The desired load was then applied compressing the spring (Fig. 6), and the test continued at the desired temperature. If failure occurs, the specimen breaks (Fig. 7 is a broken specimen) and the spring expands rupturing the strips of paper stuck on the caps, thus indicating rupture. The time of failure varies, but in the majority of cases takes place within 24 hr. after applying the load. Figure 8 shows a micrograph of a crack in a specimen which was embrittled using a boiler water.

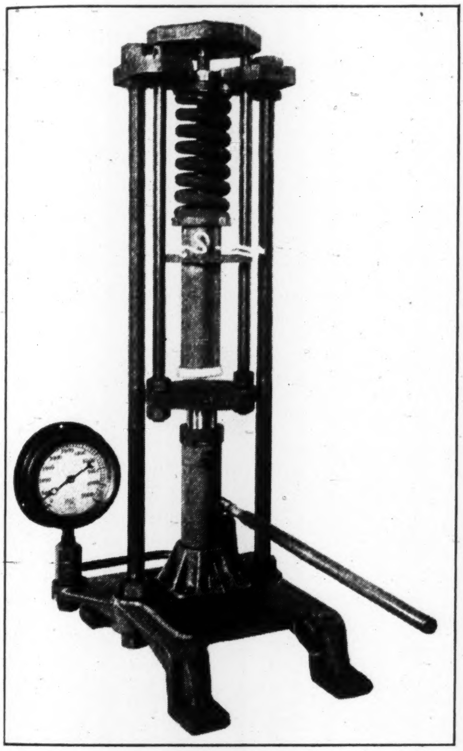

FIG. 6.—Hydraulic Press Used for Compressing Spring.

The stress, which is applied to the test specimen, was derived as the result of a series of tests in which a dilute synthetic solution of sodium hydroxide and silicate was used and various stresses ap-

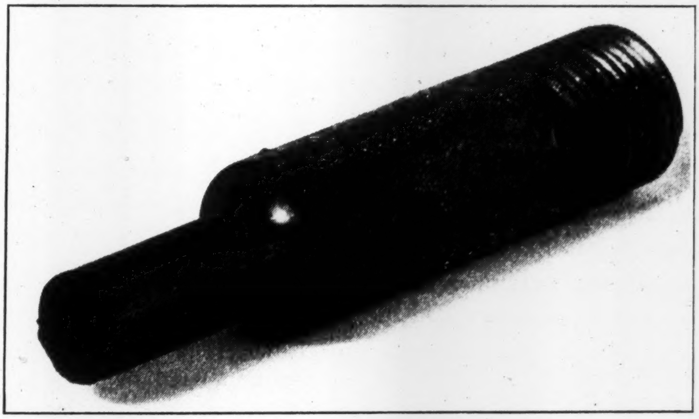

FIG. 7.—Embrittled Specimen.

plied to the test specimen at different temperatures. The stress produced by the internal steam pressure in the specimen has not been considered in the stress values given. This stress would be additional and amounts to about 1200 and 120 lb. per sq. in. at 600 and 360 F., respectively. As a result of these tests, it was found that failure resulted at temperatures between 350 and 600 F. with an applied stress of 30,000 lb. per sq. in. At 25,000 lb. per sq. in. failure did not result. It appears logical that in certain areas of the boiler, stresses above the yield point of the steel might be applied. Consequently, a stress of 40,000 lb. per sq. in. would be well above the point where embrittlement starts and still in the range of possible operating stresses. If no failure results with this high stress then the water may be reasoned to be one which has little if any embrittling tendency. If failure results and subsequent treatment prevents further failure, that treatment may be considered amply safe for boiler operation.

RESULTS OF TESTS ON SAMPLES OF BOILER WATER

The results of the tests conducted on the samples of boiler waters proved very interesting and in many instances indicated that our older ideas in regard to embrittlement were only partially correct. By assembling a representative group of tests conducted at the various temperatures, it is possible to illustrate the action of the various salts in preventing embrittlement. In order to do this, it was found advisable to assemble the results under the following temperatures:

(1) 360 and 400 F. (150 and 250 lb. pressure)
(2) 470 and 570 F. (500 and 1400 lb. pressure)
(3) 425 F. (350 lb. pressure)

Tests Conducted at 360 and 400 F.:

Tables II and III give a typical group of tests conducted at 360 and 400 F. in which no phosphate occurs. Only a few tests were conducted at 360 F.; however, a definite trend is noticeable in the results obtained. These are (1) failure results even with an A.S.M.E. ratio of 3.0, (2) sodium chloride aids materially in preventing failure. Thus, in boiler water No. 1066 increasing the ratio of the sodium chloride to the total alkalinity from 0.06 to 0.40 changed the time of failure from less than 11 hr. to 29 days when the A.S.M.E. ratio was only 0.51. When boiler water No. 1068 was tested, failure took place even with an A.S.M.E. ratio of 3.13. When the chloride content was increased so that it was 0.27 times the alkalinity, no failure resulted. In boiler water No. 1088, no failure resulted with a ratio of sodium chloride to alkalinity of 0.40 and an A.S.M.E. ratio of 0.90.

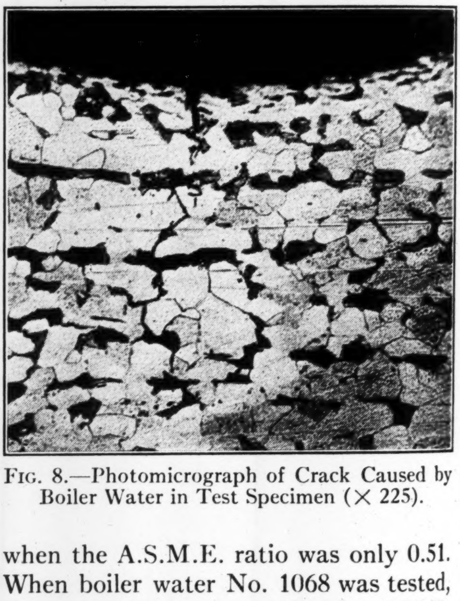

FIG. 8.—Photomicrograph of Crack Caused by Boiler Water in Test Specimen (× 225).

Table III gives the results of a representative group of tests conducted at 400 F. Here it is evident that neither the sodium sulfate nor the sodium chloride alone prevents embrittlement. However, if proper amounts of both salts are present, embrittlement may be prevented. Tests Nos. 1700, 1669, 1703, and 1715 illustrate this very clearly. When the ratio of the sodium chloride to the total alkalinity is plotted against the A.S.M.E. ratio (Fig. 9), it becomes evident that there is apparently a minimum value for the sodium chloride - alkalinity ratio (0.60) above which embrittlement is stopped even though the A.S.M.E. ratio is as low as 1.0. If this sodium chloride – alkalinity ratio is reduced below 0.60, embrittlement results even though the A.S.M.E. ratio is high. There appear to be instances where lower chloride-alkalinity ratios are effec-

TABLE II.—RESULTS OF ANALYSES OF WATERS AND OF TESTS CONDUCTED AT 360 F. (150 LB. PRESSURE).

Test	Water	Total Alkalinity, p.p.m.	Ratio, NaCl to Total Alkalinity	A.S.M.E. Ratio, Na_2SO_4 to Total Alkalinity	SiO_2, p.p.m.	Load Applied, lb. per sq. in.	Results of Tests	
							Break	No Break
No. 1759......	No. 1066....	1 410	0.06	0.51	72	45 000	0 to 11 hr.	
No. 1760......	No. 1066[a]..	1 410	0.40	0.51	72	45 000	29 days	
No. 1763......	No. 1068....	600	0.09	3.13	14	45 000	4 to 15 hr.	
No. 1766......	No. 1068....	600	0.09	3.13	14	40 000	9 days	
No. 1767......	No. 1068[a]...	600	0.27	3.13	14	45 000	....	26 days
No. 1828......	No. 1088....	10 150	0.40	0.90	395	45 000	....	29 days
No. 1852......	No. 1088[b]...	1 015	0.40	0.90	39	40 000	....	22 days

[a] Sodium chloride added.
[b] Diluted with distilled water.

TABLE III.—RESULTS OF ANALYSES OF WATERS AND TESTS CONDUCTED AT 400 F. (250 LB. PRESSURE).

No phosphate present.

Test	Water	Total Alkalinity, p.p.m.	Ratio, NaCl to Total Alkalinity	A.S.M.E. Ratio, Na_2SO_4 to Total Alkalinity	SiO_2, p.p.m.	R_2O_3, p.p.m.	Applied Load, lb. per sq. in.	Results of Tests	
								Break	No Break
No. 1620....	No. 21 630.	1 400	0.10	1.1	110	....	40 000	5 days	
No. 1623....	Synthetic.	1 400	0.10	1.1	110	0	40 000	2 days	
No. 1679....	No. 21 630[a]	1 400	0.10	2.2	110	....	40 000	4 days	
No. 1683....	Synthetic.	1 400	0.10	2.2	110	0	40 000	3 days	
No. 1718....	No. 21 630[b]	1 400	0.30	1.1	110	....	45 000	0 to 8 hr.	
No. 1735....	No. 21 630[b]	1 400	0.60	1.1	110	....	45 000	....	46 days
No. 1731....	No. 21 630[b]	1 400	5.00	1.1	110	....	45 000	....	27 days
No. 1706....	No. 1054...	1 430	1.10	9.0	124	....	45 000	....	54 days
No. 1725....	No. 1055...	795	0.25	0.4	90	....	40 000	0 to 11 hr.	
No. 1776....	No. 1071...	522	0.20	0.4	0	8	45 000	....	28 days
No. 1839....	No. 1080...	725	0.23	1.0	58	....	40 000	2 days	
No. 1905....	No. 1102	373	0.49	3.1	6	3	40 000	19 days	
No. 1954....	No. 1130...	502	0.96	2.9	9	9	40 000	....	34 days
No. 1975....	No. 1141...	316	1.13	3.1	38	5	40 000	....	30 days
No. 1976....	No. 1142...	304	1.17	3.8	25	4	40 000	....	30 days
No. 1659....	No. 1050...	1 180	0.06	1.4	180	....	40 000	0 to 12 hr.	
No. 1691....	No. 1050[b]..	1 180	0.35	1.4	180	....	40 000	17 days	
No. 1771....	No. 1069...	1 055	0.97	3.0	152	70	45 000	....	40 days
No. 1812....	No. 1084...	595	0.67	2.9	128	....	45 000	....	43 days
No. 1966....	No. 1129...	256	0.55	1.5	31	11	40 000	8 days	
No. 1965....	No. 1128...	305	0.50	1.3	21	24	40 000	7 days	
No. 1734....	No. 1061...	2 105	0.20	3.8	416	....	40 000	0 to 16 hr.	
No. 1669....	No. 1041...	1 432	0.74	0.9	230	....	45 000	....	70 days
No. 1703....	Synthetic..	1 400	0.75	0.0	230	0	45 000	1 hr.	
No. 1715....	Synthetic..	1 400	0.00	0.9	230	0	45 000	1 hr.	
No. 1700....	Synthetic..	1 400	0.75	0.9	230	0	45 000	....	54 days
No. 1953....	Synthetic..	520	0.34	0.2	2	3	40 000	0 to 16 hr.	
No. 1952....	Synthetic..	500	0.00	0.0	0	0	40 000	24 to 39 hr.	

[a] Na_2SO_4 added.
[b] NaCl added.

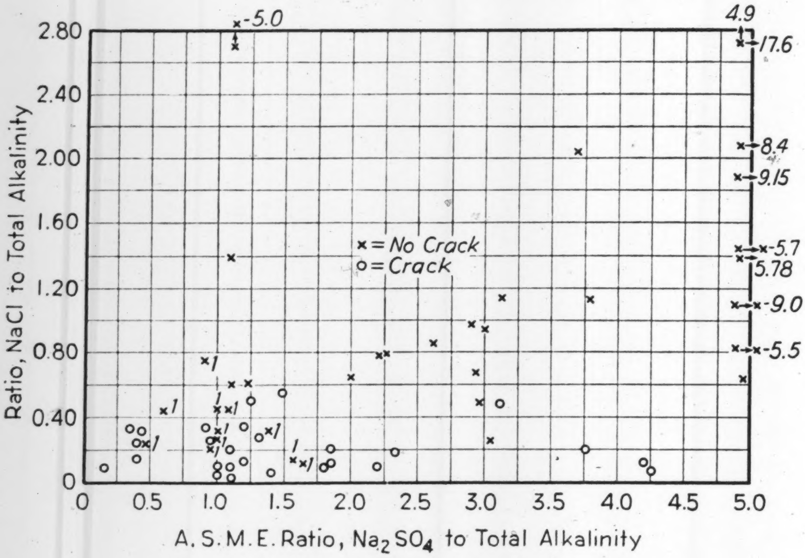

FIG. 9.—Influence of NaCl and Na_2SO_4 on Cracking at 400 F.

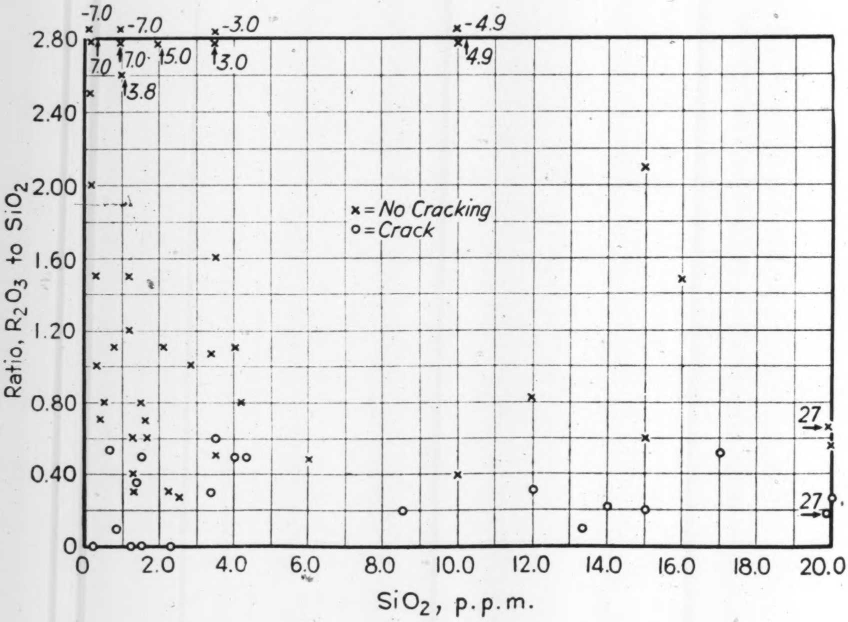

FIG. 10.—Influence of R_2O_3 and SIO_2 on Cracking at 470 and 570 F.

TABLE IV.—RESULTS OF ANALYSES OF WATERS AND TESTS CONDUCTED AT 400 F. (250 LB. PRESSURE).

Phosphate present.

Test	Water	Total Alkalinity, p.p.m.	Ratio, NaCl to Total Alkalinity	A.S.M.E. Ratio, Na_2SO_4 to Total Alkalinity	SiO_2, p.p.m.	PO_4, p.p.m.	Applied Load, lb. per sq. in.	Results of Tests	
								Break	No Break
No. 1639....	No. 1043...	1 400	0.10	1.80	306	0	40 000	8 days	
No. 1680....	No. 1043[a]	1 400	0.10	1.80	306	200	40 000	25 days	
No. 1659....	No. 1050...	1 180	0.06	1.40	180	0	40 000	0 to 12 hr.	
No. 1674....	No. 1050[a]..	1 180	0.06	1.40	180	50	40 000	2 to 16 hr.	
No. 1690....	No. 1050[a]..	1 180	0.06	1.40	180	150	40 000	11 days	
No. 1671....	No. 1033...	1 080	0.75	0.44	50	125	45 000	....	67 days
No. 1646....	No. 1045[b]..	2 450	0.18	0.01	110	160	40 000	....	29 days
No. 1650....	No. 1046[b]..	2 510	0.18	0.12	145	200	40 000	....	25 days
No. 1729....	No. 1059...	970	0.05	1.03	91	30	40 000	3 hr.	
No. 1744....	No. 1059[c]..	970	0.58	1.03	91	30	45 000	....	51 days
No. 1857....	No. 1074...	240	0.83	1.18	7	67	40 000	....	38 days
No. 1846....	No. 1076...	550	2.06	3.70	23	90	40 000	....	31 days
No. 1847....	No. 1078...	460	0.26	3.05	14	130	40 000	....	13 days
No. 1956....	No. 1132...	556	0.13	0.18	29	44	40 000	0 to 16 hr.	
No. 2004....	No. 1132[a]..	556	0.13	0.18	29	144	40 000	....	30 days
No. 1834....	No. 1086...	605	0.10	0.37	16	140	40 000	4 days	
No. 1900....	No. 1086[c]..	605	0.30	0.37	16	140	40 000	....	29 days

[a] PO_4 added.
[b] Na_2CO_3 equaled NaOH in water tested.
[c] NaCl added.

TABLE V.—RESULTS OF ANALYSES OF WATERS AND TESTS CONDUCTED AT 470 F. (500 LB. PRESSURE).

Test	Water	Total Alkalinity, p.p.m.	Ratio, NaCl to Total Alkalinity	A.S.M.E. Ratio, Na_2SO_4 to Total Alkalinity	SiO_2, p.p.m.	R_2O_3, p.p.m.	PO_4, p.p.m.	Applied Load, lb. per sq. in.	Results of Tests	
									Break	No Break
No. 1783....	No. 1076...	550	2.06	3.7	23	0	90	40 000	4 to 15 hr.	
No. 1836....	No. 1076[a]..	550	2.06	7.4	23	0	90	40 000	8 to 16 hr.	
No. 1894....	No. 1076[b]..	550	2.06	3.7	23	7	90	40 000	....	31 days
No. 1778....	No. 1075...	202	1.60	1.4	0	56	0	40 000	....	28 days
No. 1994....	No. 1130...	502	0.96	2.9	8	8	0	40 000	....	30 days
No. 1799....	No. 1083...	675	0.86	3.8	110	490	0	45 000	....	33 days
No. 1867....	Synthetic..	675	0.80	3.8	100	0	0	40 000	6 to 15 hr.	
No. 1974....	Synthetic..	675	0.80	3.8	100	200	0	40 000	....	30 days
No. 1782....	No. 1069...	1 055	0.97	3.0	152	70	0	40 000	....	27 days
No. 1781....	No. 1065...	905	0.44	2.9	84	60	0	40 000	....	40 days
No. 1778....	No. 1075..	202	1.60	1.4	0	56	0	45 000	....	28 days
No. 1890....	No. 1023...	395	0.38	0.7	60	28	85	45 000	....	23 days
No. 1778....	No. 1073...	1 280	0.12	2.0	200	60	200	45 000	0 to 10 hr.	
No. 1833....	No. 1073[c]..	1 280	0.66	2.0	200	60	200	45 000	....	25 days
No. 1787....	No. 1074...	240	0.04	0.1	7	4	67	45 000	0 to 11 hr.	
No. 1939....	No. 1074[b]..	240	0.04	0.1	7	54	67	45 000	....	44 days
No. 1889....	No. 1074[c]..	240	0.60	2.2	7	4	67	45 000	0 to 5 hr.	
No. 2002....	No. 1128[d]..	305	0.50	1.2	21	24	0	40 000	....	30 days
No. 2000....	No. 1167...	390	0.71	2.9	9	1	0	40 000	24 to 36 hr.	
No. 2018....	No. 1167[b]..	390	0.71	2.9	9	13	0	40 000	....	30 days
No. 2041....	Synthetic..	500	0.0	0.0	0	0	0	40 000	4 days	
No. 2047....	No. 1196...	403	0.85	4.0	12	4	0	40 000	14 to 17 hr.	
No. 2052....	No. 1196[b]..	403	0.85	4.0	12	10	0	40 000	....	30 days

[a] Na_2SO_4 added.
[b] Al_2O_3 added as sodium aluminate.
[c] NaCl added.
[d] Solution caused failure at 400 F. See Table III, Test No. 1965.

tive. This may be due to the fact that the effect of carbonate, undetermined organic, nitrates, etc., have not been considered. Thus it was noticeable that the cases of non-cracking in the lower chloride–alkalinity ratios, marked 1 on Fig. 9, were waters from the same power plant. Further analyses are being made of these waters in order to see if other inhibitors are present.

It is evident that in waters which have caused cracking and where the sodium chloride – alkalinity ratio has been below 0.60, increase in the sulfate content has not prevented failure, while in waters which have caused cracking and which have the A.S.M.E. ratio around 1, increasing the sodium chloride – alkalinity ratio to 0.60 or greater has stopped cracking.

The results of tests run at 400 F. using boiler waters having phosphate present are reported in Table IV. It does not appear that sufficient data are available to state definitely that phosphate does or does not prevent embrittlement. It may be stated that phosphate alone does not prevent failure. It appears possible that the presence of chloride might have a marked effect on the phosphate action. Thus in boiler water No. 1086, the addition of sodium chloride apparently made the phosphate effective. It is evident that the phosphate does not interfere with the sodium chloride – sodium sulfate combination in preventing embrittlement.

Tests at 470 and 570 F.:

Table V contains the results of a group of tests conducted at 470 F. These results show that the chloride–sulfate effect so noticeable at 400 F. has apparently disappeared at this temperature. However, embrittlement is prevented at this pressure and it appears to be due to the R_2O_3 content. The R_2O_3 content, being a combination of iron and aluminum, is a rather indefinite value. However, in the majority of the waters tested, the Al_2O_3 appeared to predominate. Tests are being conducted to study the relative effect of the iron and aluminum.

It has been shown that silica has a marked effect on the caustic action in producing embrittlement.[6] It might be considered as catalyzing the reaction. If the silica could be combined with other chemicals so as to form a complex salt, it would no longer act as a catalyst and the embrittling effect of the caustic might be stopped. In considering the action of the chlorides and sulfates in preventing failure, it has been assumed that they prevent the action of the caustic soda after having been catalyzed. Thus, relatively large amounts of these salts are necessary. If, as appears possible at the higher temperatures, the silica effect might be neutralized, the amounts of chemical necessary would bear a direct proportion to the silica. The results in Table V appear to make this assumption logical. Figure 10 shows a graph of the ratio of R_2O_3 to SiO_2 plotted against SiO_2. These results indicate that if this R_2O_3 to SiO_2 ratio is 0.6 or greater, failure has not resulted. Failure has resulted in many cases where the ratio is below this value. In many waters where cracking occurred, the addition of R_2O_3 as Al_2O_3 in sufficient amounts has prevented failure.

The tests run at 570 F. and reported in Table VI also show the effect of the R_2O_3 to SiO_2 ratio. In test No. 1993, failure resulted but the addition of 3 p.p.m. of R_2O_3 as Al_2O_3 prevented cracking. In test No. 1964, the boiler water had no free sodium hydroxide ($BaCl_2$ test) and no failure resulted even though the R_2O_3–SiO_2 ratio was low. When 100 p.p.m. of c.p. NaOH was added, failure resulted. The addition of 56 p.p.m. of Al_2O_3 along with the hydroxide prevented failure in test No. 2016.

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[6] F. G. Straub and T. A. Bradbury, "New Laboratory Data Relative to Embrittlement in Steam Boilers," *Power Plant Engineering*, February, 1936.

TABLE VI.—RESULTS OF ANALYSES OF WATERS AND TESTS CONDUCTED AT 570 F. (1350 LB. PRESSURE).

Test	Water	Total Alkalinity, p.p.m.	Ratio, NaCl to Total Alkalinity	A.S.M.E. Ratio, Na_2SO_4 to Total Alkalinity	SiO_2, p.p.m.	R_2O_3, p.p.m.	PO_4, p.p.m.	Applied Load, lb. per sq. in.	Results of Tests	
									Break	No break
No. 1815	No. 1083[a]	675	0.86	3.8	110	490	0	45 000		30 days
No. 1818	Synthetic	675	0.70	3.3	105	0	0	45 000	3 days	
No. 1910	No. 1113	520	0.34	0.2	2	3	0	40 000		28 days
No. 1993	Synthetic	520	0.00	0.0	2	0	0	40 000	0 to 5 hr.	
No. 1995	Synthetic	520	0.00	0.0	2	3	0	40 000		30 days
No. 1972	Synthetic	520	0.34	0.2	2	3	0	40 000		30 days
No. 1982	No. 1153	340	0.25	2.8	15	8	125	40 000	2 hr.	
No. 1964	No. 1139	181	0.29	0.0	18	6	54	40 000		30 days
No. 1997	No. 1139[b]	310	0.17	0.0	18	6	54	40 000	1 hr.	
No. 2016	No. 1139[c]	310	0.17	0.0	18	56	54	40 000		30 days
No. 1918	No. 1107	276	0.29	2.2	1	2	0	40 000		30 days
No. 1919	No. 1108	228	0.32	1.0	2	4	0	40 000		32 days
No. 1942	No. 1109	350	0.49	1.5	2	3	0	40 000		32 days
No. 1921	No. 1112	224	0.31	5.6	4	6	0	40 000		35 days
No. 1911	No. 1114	515	0.47	0.2	12	15	0	40 000		28 days
No. 1983	No. 1154	607	0.23	1.9	5	4	167	40 000		30 days
No. 1996	No. 1162	415	0.08	0.4	16	9	40	40 000		30 days
No. 1999	No. 1163	520	0.31	1.6	35	16	60	40 000		30 days
No. 2055	No. 1140[c]	310	0.01	0.0	0	2	57	40 000		30 days
No. 2063	No. 1140[d]	310	0.01	0.0	10	2	57	40 000	0 to 14 hr.	

[a] Test conducted at 600 F.
[b] NaOH added.
[c] Al_2O_3 and NaOH added.
[d] NaOH and Na_2SiO_3 added.

TABLE VII.—RESULTS OF ANALYSES OF WATERS AND TESTS CONDUCTED AT 425 F. (350 LB. PRESSURE).

Test	Water	Total Alkalinity, p.p.m.	Ratio, NaCl to Total Alkalinity	A.S.M.E. Ratio, Na_2SO_4 to Total Alkalinity	SiO_2, p.p.m.	R_2O_3, p.p.m.	PO_4, p p.m.	Applied Load, lb. per sq. in.	Results of Tests	
									Break	No break
No. 1902	No. 1098	533	0.44	1.5	18	6	89	40 000	0 to 11 hr.	
No. 2006	No. 1098[a]	533	0.44	1.5	18	31	89	40 000	3 days	
No. 2012	No. 1098[b]	533	0.81	1.5	18	6	89	40 000		30 days
No. 2042	No. 1191	202	0.13	3.1	10	2	0	40 000	0 to 12 hr.	
No. 2043	No. 1191[b]	202	0.62	3.1	10	2	0	40 000	5 days	
No. 2072	No. 1191[b]	202	1.10	3.1	10	2	0	40 000		30 days
No. 1917	No. 1105	330	0.18	3.9	34	9	0	40 000	4 to 16 hr.	
No. 1928	No. 1105[c]	330	0.18	4.4	34	9	0	40 000	10 days	
No. 1950	No. 1105[b]	330	0.80	3.9	34	9	0	40 000		37 days
No. 1968	No. 1145	420	0.19	3.9	22	5	0	40 000	2 days	
No. 1863	No. 1092	340	0.35	2.2	15	7	200	40 000	2 days	
No. 1886	No. 1092[b]	340	0.70	2.2	15	7	200	40 000	4 days	
No. 1855	No. 1076	550	2.06	3.7	23	0	90	40 000		34 days
No. 2011	Synthetic[d]	520	0.00	0.0	2	3	0	40 000	0 to 10 hr.	
No. 2019	Synthetic	520	0.34	0.2	2	3	0	40 000	22 hr.	
No. 2029	Synthetic	520	1.00	2.0	2	0	0	40 000		30 days
No. 1945	No. 1127	221	0.36	2.4	10	4	57	40 000	7 days	
No. 2017	No. 1127[a]	221	0.36	2.4	10	29	57	40 000	5 days	

[a] Al_2O_3 added as sodium aluminate.
[b] NaCl added.
[c] Na_2SO_4 added.
[d] Solution did not cause failure at 570 F.—see Table VI, Test No. 1995.

Boiler water No. 1140, test No. 2055, with a very low silica content but with the R_2O_3 content equal to 2 p.p.m., did not produce embrittlement even when sodium hydroxide was added. However, when 10 p.p.m. of SiO_2 was added to the same solution and tested, failure resulted in less than 14 hr. (test No. 2063). These tests show that the prevention of embrittlement at this temperature is definitely related to the silica and R_2O_3 contents.

This effect of the R_2O_3 does not appear to extend over the entire temperature range. Thus, the solution which did not cause failure in test No. 1995, Table VI, at 570 F. caused failure at 400 F. (Table III, test No. 1953). The R_2O_3 appears to be effective at 470 F. and not at 400 F., while the sulfate-chloride appears to be inactive at 470 F. but active at 400 F. In order to determine the temperature at which these salts lose their inhibiting properties, tests were conducted at the intermediate temperature of 425 F.

Results of Tests at 425 F.:

The results of a group of tests conducted at 425 F. are given in Table VII. These results indicate that the chloride-sulfate combination is effective at this temperature in preventing failure. The R_2O_3 content does not appear to be preventing failure. Thus in test No. 2019, failure resulted when using a solution which did not produce failure at 570 F. (test No. 1995, Table VI). The addition of chloride and sulfate to this solution prevented failure (test No. 2029). The addition of aluminate has not prevented failure at this temperature, whereas the addition of sufficient chloride has.

These results indicate that the chloride-sulfate effect in preventing embrittlement is active at temperatures up to 425 F. but is not effective at 470 F. The R_2O_3–SiO_2 effect is not effective at 425 F. but is active at 470 F. and above.

DISCUSSION OF RESULTS

The results of the tests conducted indicate that the method of testing boiler waters appears to give results directly applicable to boiler water treatment. Boiler water samples were obtained from seven power plants which have recently experienced embrittlement. All of these waters caused failure in the test units. Four of these waters met the A.S.M.E. ratios but had low chlorides. Two of the waters had phosphate present with low chlorides. By slight modification of these waters, they were made so as not to produce failure. Five of these plants have modified their water treatment so as to conform to the type found to prevent failure.

In several power plants operating in the 1400-lb. pressure range after having had their boiler waters tested and found to be non-embrittling even with low A.S.M.E. ratios, it was decided to operate without maintaining the A.S.M.E. ratio.

The application of the results of these tests to the control of water treatment must be done conservatively and only after a sufficient number of tests have been conducted on the type of water under consideration. A test run with the new testing unit is a severe one. The conditions are such that they might not always be encountered in operation and if a water were to produce embrittlement in the test, it does not follow directly that the boiler using this water would be embrittled. However, if a water produces failure in these tests and a slight modification of the boiler water prevents failure, then it is safe to predict that the modified water would be an extremely safe type to use so as to be certain that the boilers will operate free from embrittlement.

The addition of aluminate to prevent embrittlement at the higher temperatures should only be started after careful consideration of all the factors involved. In certain installations, the use of small amounts of sodium aluminate appears logical while in others it might cause other troubles. Any reduction in the silica at the higher pressures would aid in raising the R_2O_3–SiO_2 ratio and thus prevent embrittlement. In many of the higher pressure plants, it is being found that there is a natural balance of these two chemicals which makes the water non-embrittling in nature without any special treatment for the prevention of embrittlement.

Arrangements have been made whereby the Chemical Engineering Department of the University of Illinois will test boiler waters for any interested parties. These tests will involve analyses of the waters and testing samples in the new embrittlement testing units. A nominal charge will be made for this work to cover the cost of conducting the analyses and experiments. Any profit accruing as a result of these tests will be used by the department for further research on boiler water problems of general interest to the steam power plant operators. From time to time the results of these tests will be published. In this manner, it will be possible to assemble these data at a central disinterested laboratory and at the same time answer the question whether any particular boiler water is embrittling or not for the power plant operator. Of course, the identity of the source of the samples tested will not be revealed.

CONCLUSIONS

The conclusions reached from the data obtained so far may be summarized as follows:

1. A new method of testing samples of boiler water to determine whether or not they will cause embrittlement has been developed.
2. For steam pressures up to 250 lb. per sq. in. embrittlement may be prevented by maintaining the sodium-chloride content of the boiler water greater than 0.6 times the total alkalinity expressed as sodium carbonate along with the sodium sulfate content greater than 1.0 times the total alkalinity.
3. For steam pressures between 500 and 1400 lb. per sq. in., these results would indicate that the presence of a soluble R_2O_3 content of greater than 0.6 times the SiO_2 content of the boiler water prevents embrittlement.
4. For a steam pressure of 350 lb. per sq. in., the sulfate and chloride to alkalinity ratios appear to be effective in preventing embrittlement, although larger amounts may be necessary than at the lower pressures.

DISCUSSION

Mr. Everett P. Partridge[1] (*presented in written form*).—The use of sodium sulfate as an inhibitor of "caustic embrittlement" first received official recognition twelve years ago, when recommendations for the maintenance of certain ratios of sodium sulfate to alkalinity were first written into the A.S.M.E. Boiler Code. In the period since 1926, these recommended ratios have attained a quasi-legal status. In spite of the skepticism voiced generally by engineers in other countries, and in the United States chiefly by the engineers responsible for water treatment on the railroads, millions of pounds of sodium sulfate or of sulfuric acid have been added to boiler feedwaters, in the hope that embrittlement would thus be prevented.

In many cases, maintenance of the sulfate-alkalinity ratios means either that the total concentration of dissolved salts in the boiler water must increase, or that more water must be blown down from the boiler, in order to hold a predetermined level of concentration. The operator has therefore been faced with the choice between possible penalty if he disregarded the ratios, or of wet steam or of increased costs or both, if he adhered to them.

When a move was made in 1929[2] to change the ratio value to 0.014 times the steam pressure for boilers in the range from 150 to 600 lb. gage pressure, such strong opposition developed that the original values of 1 for pressures up to 150 lb., 2 in the range from 150 to 250, and 3 for pressures above 250 lb. were retained. Since the proposed change would have required the operator to maintain a concentration of sodium sulfate 8.4 times the total alkalinity in a boiler operating at 600 lb. pressure, this reaction was scarcely surprising.

Five years ago, when a new investigation was undertaken cooperatively by the Joint Research Committee on Boiler Feedwater Studies and the U. S. Bureau of Mines, the A.S.M.E. ratios were so firmly established that the program agreed upon was a study of the solubility relations of sodium sulfate in the saline solutions which would be attained by the excessive concentration of boiler waters of various compositions. The statement was even made during the preliminary discussion of the work that everything worth while had already been done, and further research was merely a waste of time and money. It was only because the solubility studies, when undertaken, took less time and money than had originally been anticipated that some further exploration of the mechanism of embrittlement was commenced in 1934.

How far we have come in the four years since the embrittlement question was re-opened can only be appreciated when we look back and consider the successive steps. In December, 1934, the first tests reported were at variance with Mr. Straub's earlier data, in that stresses much above the room temperature yield point were found necessary to produce failure when steel specimens were subjected to contact with concentrated solutions of pure sodium hydroxide at 250 C. By 1935 the apparent discrepancies had accumulated to such extent that every item in the experimental technique in the respective investigations at Urbana and at New Brunswick was being scrutinized under the supervision of the subcommittee of the Joint Research Committee responsible for the program. The answer was found in the presence of one or two tenths of one per cent of silica in the technical grade of caustic soda used by Mr. Straub, as against the very much smaller amount of this constituent present in the reagent grade caustic soda used by Mr. Schroeder.[3]

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[1] Director of Research, Hall Laboratories, Inc., Pittsburgh, Pa.

[2] *Mechanical Engineering*, Vol. 51, p. 88 (1929).

The discovery of the role played by silica in promoting intergranular attack on steel by caustic solutions led to further study which demonstrated that the type of failure characteristic of "caustic embrittlement" could be produced in the laboratory by a variety of solutions, even including dilute nitric acid. In every case the mechanism seemed to be that of corrosion limited to the grain boundaries by the combined effects of an attacking agent and a protective agent, which stopped the reaction of the attacking agent with the grains themselves, but thereby accelerated its penetration along grain boundaries.[4]

That embrittlement is much less of a settled question now than it appeared to be five years ago is confirmed by the present paper, in which sodium sulfate by itself is now found to be ineffective as an inhibitor, although when reinforced by sodium chloride it apparently prevents cracking over the limited pressure range up to 350 lb. gage. These conclusions well illustrate the fact that every investigator, if he works sufficiently long in a particular field, will be forced to reverse some of his earlier judgments. For example, Mr. Straub once found[5] during laboratory tests at 500 lb. gage pressure that cracking was prevented when the ratio of sodium sulfate to alkalinity was 3.6, although rapid failure occurred when the value was 2.8 or lower. It is not entirely clear from the context whether sodium chloride was also present in the test solution, although the statement is made, "If sodium chloride is also introduced, then the best conditions for cracking are obtained, and if the sulfate can stop embrittlement under this ideal condition, the conclusion can be drawn that the ratios obtained are protective."

The new method of testing described in this paper ingeniously eliminates the gap that once existed between the low concentration of dissolved substances present in actual boiler waters and the high concentration required to produce intergranular cracking in steel in the laboratory; by using a hollow test specimen with a closely fitting filler, the constituents present in the initial samples of boiler water are apparently concentrated to a high degree in the annular space between the filler and the test specimen. The mechanism by which the concentration of sodium hydroxide in the water above the filler could be reduced from 800 down to 60 p.p.m. during a test is, however, obscure. One would scarcely expect a dissolved substance to diffuse continuously from a region of lower concentration to a region of higher concentration. The most likely explanation seems to be a process of distillation of water vapor from the minute annular space around the filler to the larger space above it, followed by replacement with solution. Repeated variations in pressure within the test specimen conceivably would cause this process to continue until relatively concentrated solution had been developed in the small annular space. This process would be quite analogous to that previously observed by Mr. Straub in glass apparatus at the atmospheric boiling point.[6] The basis for the necessary variation in pressure in the new apparatus may be found in the fact that the test temperatures were controlled only to ± 15 F. If the actual temperature of the test specimen fluctuated between the extreme limits, the pressure within the specimen at a nominal test temperature of 360 F. would change by nearly 60 lb., while at the highest test temperature of 570 F. the corresponding change in pressure would be 290 lb. It is interesting to speculate whether embrittlement would be produced in this apparatus if the test specimens were held at a closely controlled temperature.

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[3] W. C. Schroeder, A. A. Berk and Everett P. Partridge, "Effect of Silica on the Failure of Boiler Steel in Tension in Contact with Sodium Hydroxide Solution," Progress Report No. 8, October 1, 1935 (unpublished).

W. C. Schroeder and A. A. Berk, Am. Inst. Mining and Metallurgical Engrs., *Technical Publication 691*, 14 pp. (1936); also in *Combustion*, Vol. 7, pp. 29-33 (1936).

F. G. Straub and T. A. Bradbury, *Power Plant Engineering*, Vol. 40, pp. 104-5 (1936).

[4] W. C. Schroeder, A. A. Berk and C. H. Fellows, "Intercrystalline Cracking of Boiler Steel," *Journal*, Am. Water Works Assn., Vol. 30, pp. 679-694 (1938).

[5] F. G. Straub, "Embrittlement of Boilers," *Bulletin 216*, p. 71, Engineering Experiment Station, University of Illinois (1930). Reprinted, 1935.

The general validity of the conclusions reached in this paper would be somewhat impaired if the test method is fundamentally dependent upon a rather wide variation in pressure. There is also the additional difficulty that unless the restricted space between the upper part of the filler and the wall of the specimen is reproduced very precisely from test to test, the concentration produced below this restriction might vary over rather wide limits.

With respect to the use of alumina as an inhibitor of embrittlement in boilers operating at high pressures, a word of caution should be shouted rather than whispered. High-pressure boilers have been characterized by a tendency to form sodium aluminum silicate scales whenever the necessary constituents were present in the boiler water. One case may be cited in which a boiler operating at 600 lb. pressure, with 100 per cent make-up, suffered tube failures due to sodium aluminum silicate scale. A check on the feedwater showed that the alumina content varied between 1 and 2 p.p.m. Removal of this alumina has obviated further difficulty.

Because the lignin and tannin bodies seem to have a very specific effect as inhibitors of embrittlement[7] it is to be hoped that Mr. Straub will study their effect in his test apparatus and will report his findings in the near future. In the meantime the boiler operator is left with the responsibility of choosing between maintenance of the original A.S.M.E. embrittlement ratios, which are supported by the experimental findings of neither Straub nor Schroeder; of adopting the modified ratios proposed by Straub in this paper; or of using organic inhibitors of the lignin or tannin class, as suggested by the work of Schroeder. What the operator should do will rest largely upon the further results from the respective investigations.

MR. E. B. POWELL[8] (*presented in written form*).—The authors present a new and attractive theory of the capillary space in its promotion of the high degree of boiler water concentration which has seemed necessary for the embrittlement of boiler steel. The more conventional theory is explored and its development also accompanied by substantiating experimental data in the article by Messrs. Schroeder, Berk, and O'Brien appearing in the June issue of the *Bulletin* of the American Railway Engineering Association. Both theories have logical support. Neither would seem at this time to have had sufficiently exhaustive investigation to furnish an adequate basis for the drafting of embrittlement preventive formulas. Possibly, the complexities of natural waters may long delay the development of rational formulas of universal application in safeguarding steam boilers against embrittlement. In any case, in the interval of the formulation of an adequately comprehensive theory and in the present seeming conflict of ideas on chemical actions involved, apparatus in which the individual plant's boiler water could be tested for embrittling tendencies, and appropriate correctives determined if needed, would be a great boon in meeting the situation temporarily.

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[6] F. G. Straub, "Embrittlement of Boilers," *Bulletin 216*, pp. 82-85, Engineering Experiment Station, University of Illinois (1930). Reprinted, 1935.

[7] W. C. Schroeder, A. A. Berk and R. A. O'Brien, "Protecting Steel Against Intercrystalline Attack in Aqueous Solution," *Transactions*, Am. Soc. Mechanical Engr., Vol. 60, pp. 35-42 (1938).

[8] Consulting Engineer, Stone & Webster Engineering Corp., Boston, Mass.

In going over the data now presented by Messrs. Straub and Bradbury, one is struck by their departure from previously published results, their seeming contradiction, in fact, of results reported by numerous investigators working with apparatus of different design. Possibly, the present authors may have secured the nearest approximation in simulating the functioning of the capillary space in the embrittled commercial boiler. Possibly, evaporation may play a more important part than they now assume, or it may be the controlling factor in some instances. The vital importance of the questions which arise warrants an exceedingly careful re-examination of other related research and of instances of commercially encountered embrittlement, in so far as available evidence may permit, as well as close critical analysis of the present test unit.

I think it will be granted that a test unit which is to be used for direct development of embrittling tendencies of water under particular conditions should either reproduce those conditions essentially or depart from them only in aspects clearly recognized and definitely evaluated. Unfortunately, our knowledge of the actual mechanism of embrittlement of steel in the commercial boiler is far from complete or satisfactorily clear. However, in considering critically the authors' test unit, the following have seemed to need evaluation in the light of such non-controversial data as can be brought to bear upon them:

1. *Temperature Distribution.*—The test unit seems designed with great care to give uniform temperature conditions in the specimen. It would be of interest to know the actual temperature rise in the lower part of the specimen, where the diameter is reduced, in the vicinity of the typical fracture, in relation to the temperature indicated by the thermocouples shown in Figs. 1 and 3.

2. *Oxidation of Filler.*—The filler used in forming the capillary space within the specimen is described as oxidized prior to use. The presence of metals of distinctly different surface conditions in contact with a fairly strong electrolyte suggests the possibility that the e.m.f. promoted by such difference in surface conditions might be an important factor in the corrosion and embrittling effects observed. Possibly this is actually a factor of significance in the typical capillary space of the embrittled commercial boiler.

3. *Stress Application.*—Application of stress after attainment of full temperature in the metal and solution under test, while doubtless in some degree characteristic of the locomotive boiler operation, would seem highly unusual in the stationary steam generating unit.

The records of the American high-pressure boiler plants would indicate that many of these are operating with $R_2O_3 - SiO_2$ ratios much lower than that chosen by the authors as preventive of embrittlement. I agree with the authors emphatically that there should be no attempt at indiscriminate increase in R_2O_3 content of these high-pressure boiler waters but that, at least, the seriousness of the hazard of analcite scale formation with the particular water and operating conditions of the individual plant should first be determined. Consideration of the authors' data suggests that those boilers of the 1200 to 1400 lb. class in which the chemical conditions which they have found inhibitive do not obtain in the boiler water may owe their immunity from embrittlement to the stress relieving of the drums and a generally higher standard of workmanship employed in their fabrication throughout.

A point on which all of our embrittlement research seems to be in agreement, and which gains much support from experience with the commercial boiler, is the importance of a highly stressed condition in the metal as a factor in causing embrittlement. Apparently, not only must the chemical conditions of the water be favorable to embrittlement but it is no less essential that the metal in contact with this water be subject to extremely high stress if embrittlement is to take place. Thus, it appears that boiler design and manufacture control the basic conditions which make possible or, with reasonable definiteness, avoid the subsequent embrittlement of the steel. Present seam welding and drum stress relieving practice probably represents the most positive single step so far taken toward embrittlement prevention. The occasion seems opportune for a plea to the boiler manufacturers so to develop their methods of tube expanding as to give definite assurance of uniform freedom from possibility of capillary space or of stress sufficient to encourage embrittlement, and that, having attained this accuracy of method, they also devise inspection procedure which will afford clear evidence of the tube joint condition obtaining in the commercial boiler as it will be then fabricated.

MR. J. B. ROMER[9] (*presented in written form*).—The test data presented by the authors cover a wide range of conditions and indicate that boiler metal cracking or caustic embrittlement, as it is sometimes termed, can be inhibited by the proper selection of inhibiting agents.

Boiler metal cracking has been an active question for many years and has been studied from many angles. Many of the early laboratory investigations were conducted in glass vessels with strong caustic solutions, and hence any effect that silica may have had was entirely obscured. Many of the early investigators felt that atomic hydrogen played a part and they supplied the necessary atomic hydrogen by the application of an external e.m.f. In other experiments, sealed, steel bombs were placed within the drums of operating boilers and certain experimental boilers were built and operated. The available literature was studied and the workers were constantly in touch with field experiences, all in an effort to obtain a rational explanation of this type of under-water cracking. The valuable data and experiences, thus collected, led to the formulation of the A.S.M.E. ratio for the prevention of embrittlement.

Our organization has always been interested in this specific problem and had a very active part in the studies which led to the adoption of the present-day ratios by the A.S.M.E. Subcommittee on the Care of Power Boilers in Service. The maintaining of this ratio has saved boiler operators many thousands of dollars; of this, there is no question.

In the last ten to fifteen years, boiler construction has materially changed.

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[9] Chief Chemist, The Babcock & Wilcox Co., Barberton, Ohio.

Such riveted construction as has been fabricated has been inside caulked; welded construction has eliminated the butt-strap and other types of seams which were the locations wherein most of this type of cracking developed.

Operating pressures today are much higher than they were when the ratios were first adopted.

These conditions may require a new study to determine the utility of existing inhibiting materials and whether or not more beneficial materials are available for this purpose.

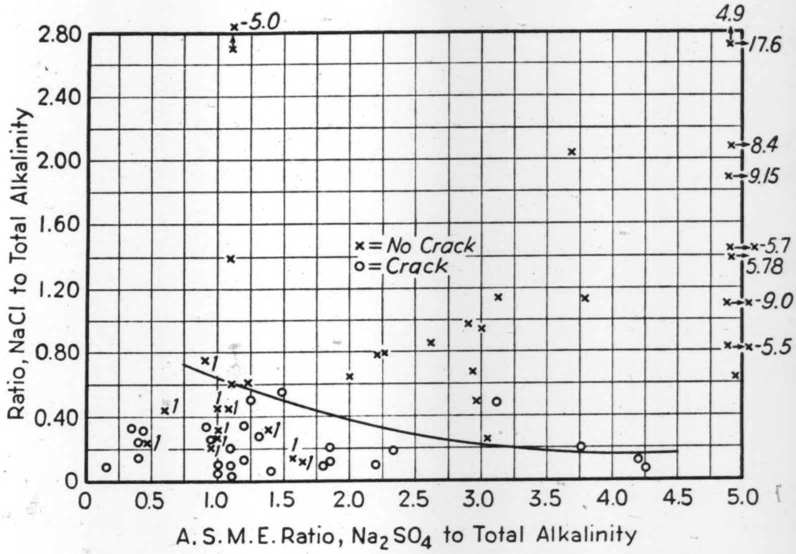

FIG. 1.—Influence of NaCl and Na_2SO_4 on Cracking at 400 F.; Pressure, 250 lb. Absolute.

The test unit, described by the authors, has a feature which ties it in with past boiler experiences to a greater degree than any previous test equipment. This feature is the small annulus, or capillary space between the test specimen and the filler bar, and the even smaller capillary space at the top of this filler. Although the authors use boiler waters or solutions of comparable concentrations, they are able to concentrate these solutions in this capillary space in a manner distinctly analogous to the action within the seams of a boiler. This is a feature never heretofore obtained simultaneously with the control of temperature and pressure.

Figure 9 of their paper shows the influence of sodium chloride and sodium sulfate on cracking at 250 lb. absolute, or 450 F. In order to bring out certain points, this figure has been reproduced in the accompanying Fig. 1. The cracking, with three exceptions, occurred at, or to the left of, the ordinate representing the A.S.M.E. ratio of 3.0. This is the minimum ratio recommended by the code for this pressure. It will be noted further that when the water tested had a low sulfate ratio, a higher sodium chloride ratio was necessary in order to prevent cracking.

It is unfortunate that the authors have not, as yet, had an opportunity to explore more fully the area where the A.S.M.E. ratio is less than 1.0, as such an exploration might substantiate the thought that the amount of sulfate required increases as the amount of sodium chloride decreases and *vice-versa.*

While the line of demarcation between cracking and no cracking is not clearly defined, its apparent location is shown in Fig. 1.

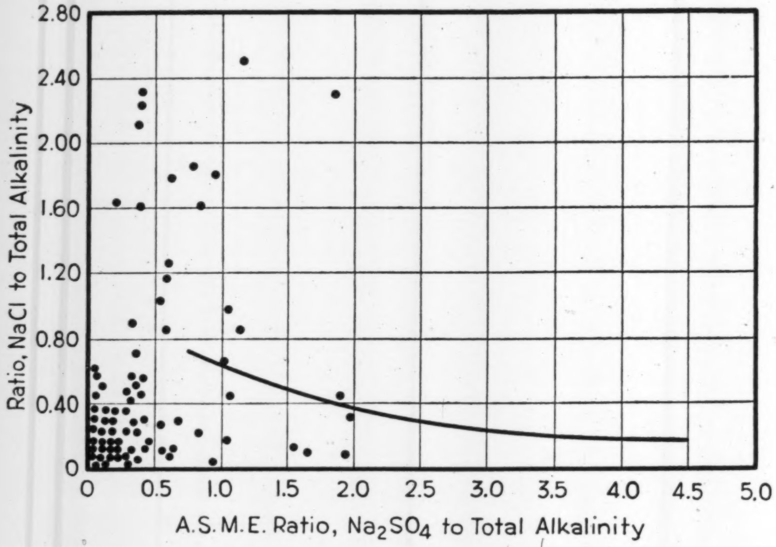

FIG. 2.—Influence of NaCl and Na_2SO_4 on Cracking Service Record of Failures. Pressure, 100–250 lb. Absolute.

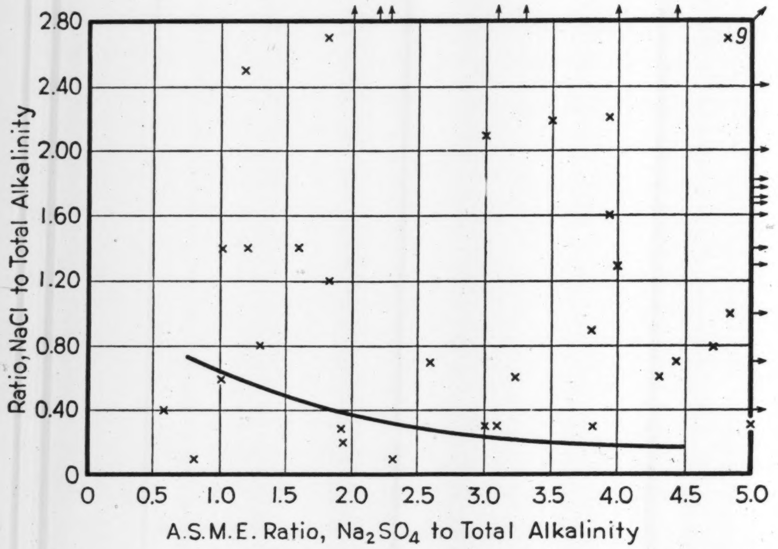

FIG. 3.—Inhibiting Influence of NaCl and Na_2SO_4 Service Record, No Failures. Pressure, 100–250 lb. Absolute.

A question which invariably arises in every case of laboratory research is "What is the correlation between laboratory and plant?" To aid in answering this question, we have reviewed the data from every case of this type of failure where a complete record was available. These cover the pressure range from

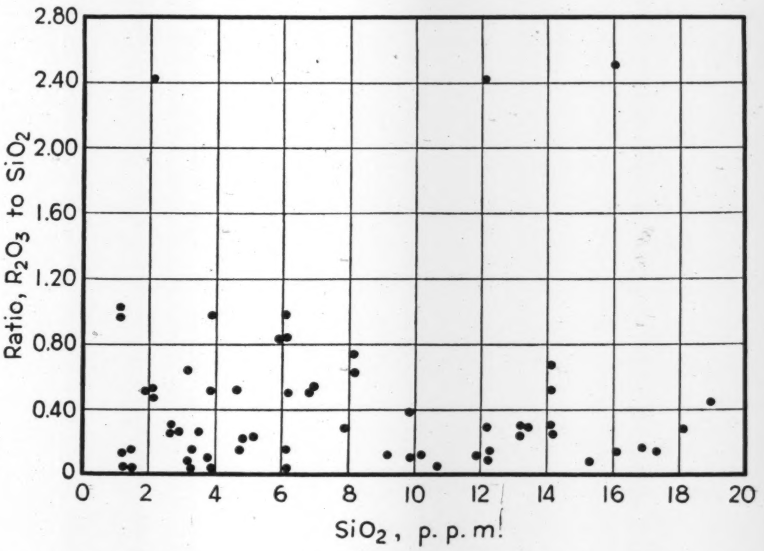

FIG. 4.—Influence of R_2O_3 and SiO_2 on Cracking Service Records of Failures. Pressure, 100–250 lb. Absolute.

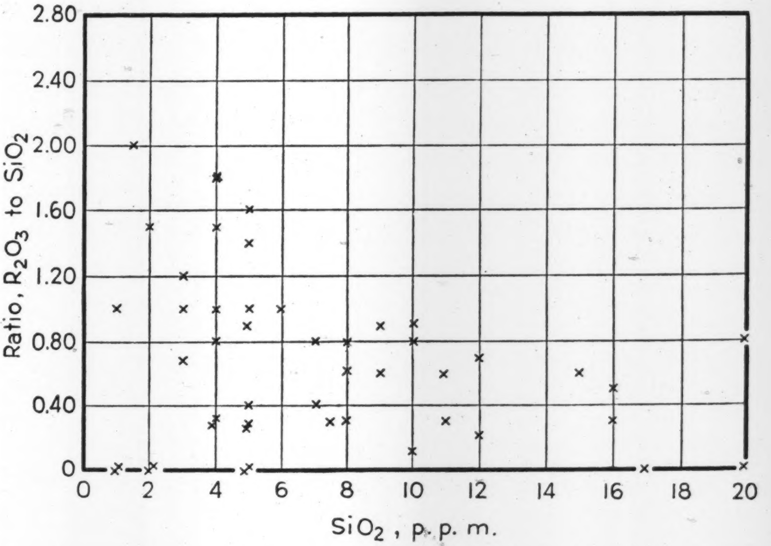

FIG. 5.—Inhibiting Influence of R_2O_3 and SiO_2 Service Record, No Failures. Pressure 100–250 lb. Absolute.

100 to 250 lb. absolute. It is extremely interesting to note, from the accompanying Fig. 2 that every one of these points is at, or to the left of, the ordinate representing the A.S.M.E. ratio of 3.0. If we draw a line similar to that drawn in Fig. 1, we find that 78 per cent of all failures are to the left, or below, this line. A great many of those above the line are in a region which Messrs. Straub and Bradbury have not as yet explored.

We have records of boiler-water conditions covering many jobs where boiler metal cracking of the type under discussion has not, to our knowledge, occurred. Data on these are shown on the accompanying Fig. 3. Few cases are below this line. A large number of the cases are beyond the limits of the coordinates shown and are indicated by the arrows. It should be noted that these records cover the same period of time as those previously discussed and in no case do they cover internally caulked, seamless-forged or welded drums.

The data of Figs. 2 and 3 afford corroboration of the existing code ratio.

Messrs. Straub and Bradbury show in Fig. 10 the influence of R_2O_3 and silica on cracking at 470 and 570 F., corresponding to 515 and 1225 lb. absolute. Their data do not show any failures when the R_2O_3-silica ratio is above about 0.6; however the ratios, particularly when the silica content is low, fail to produce cracking in some cases.

Although we do not have sufficient service records at these temperatures and pressures, we do have them in the 100 to 250 lb. range. In the accompanying Fig. 4, we are showing our data with reference to R_2O_3 and silica in cases where failures occurred in the pressure range 100 to 250 lb. The corresponding data in cases where failure did not occur are shown in the accompanying Fig. 5. Although the plotted points in these two figures cover different areas, there is a distinct tendency for overlapping, much more so than was noted in the sulfate and chloride relation.

It is to be noted that most of the failures occurred when the R_2O_3 and silica ratio was below about 0.6 or 0.8, whereas the cases where failure did not occur were, for the most part, where the R_2O_3-silica ratio was above about 0.4.

MESSRS. W. C. SCHROEDER[10] AND A. A. BERK[10] (*by letter*).—The testing unit described in this paper makes it possible to secure a reduction in the load-carrying ability of a steel specimen through the action of a boiler water without resorting to concentration of the water by the use of intermediate control devices, or in a separate vessel. It may, therefore, offer a closer approach to some of the conditions that act to produce intergranular or embrittlement cracking in boiler steel.

This unit eliminates some of the assumptions involved in the earlier tests for intercrystalline cracking, but on the other hand several new assumptions must be made to relate the results obtained in it to boiler operation. Some of these are:

1. That a boiler water concentrates, or by some mechanism produces cracking, in a small space communicating only with the interior of the boiler. It will be shown in the discussion that intercrystalline cracking can be produced as well by very slow leakage of the boiler water toward the atmosphere.

2. That the volume of solution in the test unit is sufficient to produce the same conditions as produced by the large volume of water in a boiler. This point seems especially important since use of several liters of solution might allow a greatly increased concentration of sodium hydroxide in the space where

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[10] Chemical Engineer, and Assistant Chemist, respectively, Eastern Experiment Station, U. S. Bureau of Mines, College Park, Md.

cracking occurs. It has already been shown that the weak protective action of some compounds, for example sodium sulfate, may be greatly reduced or destroyed by increase in sodium hydroxide concentration.

3. That the size or shape of the capillary space between the filler and the specimen used in tests to show that certain substances inhibit cracking does not influence the results. The spaces where concentration may take place in a boiler are not machined to size and the space may well determine the extent to which concentration may occur.

4. That contact of the boiler water with the fresh metal surface of the specimen in the upper chamber does not materially change the composition of the sample. The validity of this assumption is subject to some doubt. For example, a solution containing 500 p.p.m. sodium hydroxide and 50 p.p.m. of sodium silicate has been found to lose as much as 40 ppm. of sodium silicate when heated in contact even with an oxidized metal surface. Similarly, oxygen introduced into the sample in transfer from the boiler to the test unit may destroy organic matter or other oxidizable compounds, or may materially influence the character of the oxide surface and the possibility that cracks will or will not result during the test. This means that the solution undergoing test in the unit is not necessarily the same solution that was withdrawn from the boiler.

5. That if the boiler water mechanically plugs the annular space in the test unit to prevent embrittlement it would, therefore, plug all the gaps in the boiler to prevent embrittlement. Whether or not plugging prevents cracking in the test unit might be determined by examining the space between filler and specimen at the end of the test. On the other hand, if the application of heat plays a part in the concentration of the solution it may well play a part in the formation and maintenance of the plugging effect, and examination at room temperature at the end of the test would not necessarily reveal conditions that existed at higher temperature.

All of these assumptions led to further interesting speculation, but time is too brief to discuss them in detail. The writers have, however, started out with quite a different view of the mechanism that causes concentration of the boiler water and cracking in the metal, and it is interesting to see the large difference in results produced by this new viewpoint.

Concentration Mechanism.—At the edge of a butt strap or rivet head it is sometimes possible to observe deposits of white solid, which frequently consist of the soluble salts such as sodium sulfate and sodium chloride. These materials can only deposit from the boiler water after it has concentrated many hundredfold, and the mechanism might be pictured as a very slow diffusion of the boiler water past the edge of the butt strap and rapid loss of steam once it has reached atmospheric pressure.

During this process the dilute boiler water steadily increases the concentration of all its dissolved solids until they reach saturation and begin to crystallize from solution. If sodium hydroxide is present, the solution may easily pass through the concentration range that will cause intergranular cracking.

At the edge of a butt strap it is doubtful whether the metal would be sufficiently cold worked or stressed so that cracking would occur. If concentration occurs down in a riveted seam, a different situation exists, and contact of the concentrated solution with the cold-worked metal could cause cracking.

The rate of leakage must be confined within certain limits to produce concentration by this mechanism. If no leakage occurs, concentration is not possible. On the other hand if the leak is too large, water will rush out so rapidly that evaporation will be far too slow to allow concentration. The most dangerous type of leakage is that which would allow an almost imperceptible loss of water vapor to the atmosphere.

One test has been reported on a locomotive boiler to see whether diffusion of steam from a riveted seam toward the atmosphere could be detected. The boiler was heated to operating pressure and cold mirrors were passed slowly along the seams. In several places leakage of steam was occurring at sufficient speed to produce water on the mirror surface. More tests of this character should be carried out, although a leak even below the limits of sensitivity of this test might be dangerous.

In the laboratory, at 200 lb. pressure, it has been possible by this process to concentrate boiler waters containing from 500 to 2000 p.p.m. of sodium hydroxide to solutions containing more than 600,000 p.p.m. of sodium hydroxide.

Tests with Sodium Sulfate - Sodium Chloride Solutions.—By the use of this concentration principle involving the slow diffusion of the dilute boiler water past a restriction and evaporation on the low-pressure side of the restriction, it has been possible to construct equipment that will allow the direct testing of the water for its action in producing intercrystalline cracks. The conditions existing during this test would appear about the same as those existing during the slow leakage of boiler water through a riveted seam toward a region of lower pressure. As much solution can be passed through the equipment as desired and control is provided over the rate of leakage.

Two tests were run in this equipment using water alone and with a 60,000 lb. per sq. in. spring stress failure did not occur in 10 or 12 days at 130 lb. gage. These tests show that at this or lower stresses failure cannot be produced by mechanical factors alone. With sodium hydroxide - sodium silicate solutions, failure was produced in 1 to 4 days at this temperature with spring stresses from 30,000 to 55,000 lb. per sq. in. The cracking was intercrystalline.

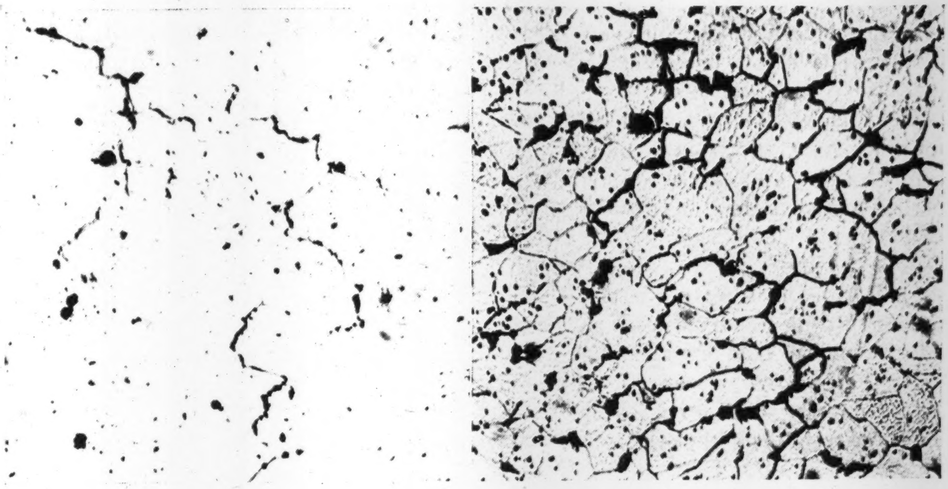
The accompanying Table I shows the results of tests in this equipment with sodium hydroxide - sodium silicate solutions also containing sodium sulfate and sodium chloride. Failure was encountered in every case, and the failure time was not measurably different from that found in similar solutions in the absence of sodium chloride. The first test solution contained sodium chloride but no sodium sulfate. The sodium chloride did not influence the failure time. In tests Nos. 38 and 74, the ratios of sodium sulfate and sodium chloride to total alkalinity were 1.5 and 0.46, respectively. Failure occurred in both cases although one specimen did run for several days longer than normal time. The last four solutions, all containing sodium sulfate

TABLE I.—FAILURE OF SPECIMENS IN THE CONCENTRATION TESTS IN SOLUTIONS TREATED WITH SODIUM SULFATE AND SODIUM CHLORIDE.

Specimens of cold-rolled steel. Temperature 180 C. (130-lb. gage). Spring stress 45,000 lb. per sq. in.

Specimen	Water Composition, p.p.m.				Test Result
	$NaOH$	Na_2SiO_3	Na_2SO_4	$NaCl$	
No. 41	500	25	......	500	broken 3 days
No. 38	500	25	1000	300	broken 11 days
No. 74	500	25	1000	300	broken 2½ days
No. 37	500	25	1000	500	broken 1 day
No. 54	500	25	1000	500	broken 1 day
No. 79	500	25	1000	1000	broken 1½ days
No. 40	500	25	1000	2000	cracked 4 days[a]

[a] Specimen so badly cracked when examined at end of this time that reloading was unnecessary.



Unetched Etched

FIG. 6.—Intercrystalline Cracks Produced by Action of a Sodium Hydroxide - Sodium Silicate Solution Containing Sodium Sulfate and Sodium Chloride (× 300).

COMPOSITION OF SOLUTION

500 p.p.m. NaOH........................ 1000 p.p.m. Na_2SO_4
25 p.p.m. Na_2SiO_3...................... 1000 p.p.m. NaCl

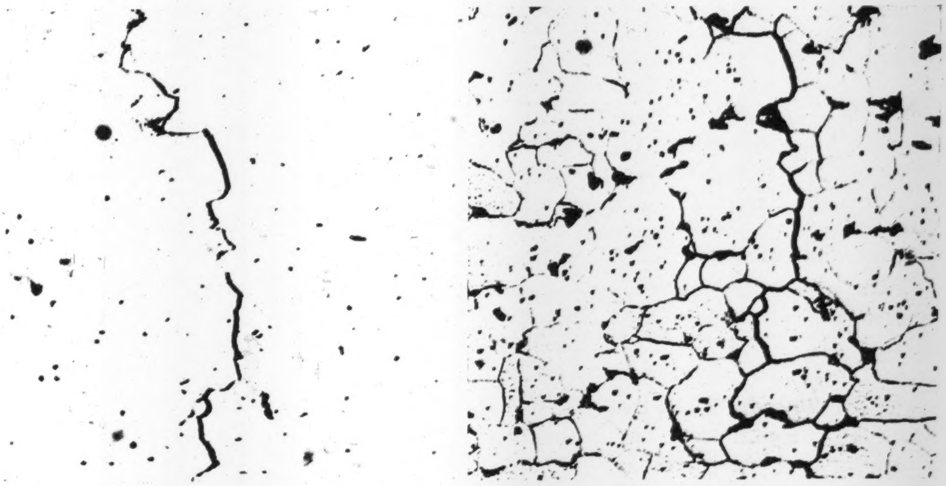


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FIG. 7.—Intercrystalline Cracks Produced by Action of a Sodium Hydroxide - Sodium Silicate Solution Containing Sodium Sulfate and Sodium Chloride (× 300).

COMPOSITION OF SOLUTION

500 p.p.m. NaOH........................ 1000 p.p.m. Na_2SO_4
25 p.p.m. Na_2SiO_3...................... 2000 p.p.m. NaCl

and sodium chloride to total alkalinity ratios considerably greater than 1.0 and 0.6, respectively, were broken or severely cracked in less than 4 days.

Cold-rolled steel specimens were used for all the tests shown in Table I.[10a] Intercrystalline cracks are found in areas of boiler metal that have suffered cold work during fabrication. For example, the metal under rivet heads, along the rivet holes, and the rivet itself are cold worked during construction of the boiler. The tubes are cold worked during rolling. These areas are usually the ones where intercrystalline cracking starts. To these residual cold work stresses may, of course, be added any applied stress resulting from steam pressure or other factors. It seems necessary that a chemical treatment prevent embrittlement in the presence of cold work stress if it is to be effective in a boiler. The sodium sulfate and sodium chloride do not fulfill this requirement.

The accompanying Figs. 6 and 7 show the type of cracking produced in two of the specimens in the solutions containing sodium sulfate and sodium chloride. It is almost completely intercrystalline, and the photomicrographs certainly will not support the belief that these salts will prevent intercrystalline cracking in steel. The photomicrographs are cross-sections of the specimens and they do not indicate the elongation of the crystals resulting from the use of cold-rolled steel as much as would a longitudinal section.

Further support for these results is also available from tests in the regular tension testing equipment in which the concentrated solution is placed directly in contact with the specimen. The accompanying Table II shows that failure was not prevented at any of the test temperatures in spite of the addition of considerably more sulfate and chloride than was necessary to satisfy the ratios of 1.0 and 0.6.

The history of a 385-h.p. 3-drum boiler operating at 100 lb. pressure is also of considerable interest with regard to the action of sodium sulfate and sodium chloride in preventing embrittlement. In its first three years of operation this boiler cracked about 200 water circulating tubes at seats in the upper drums. The drum metal at the tube seats was also cracked, and in the second year one drum was replaced because cracking had progressed too far into the tube ligaments. The new drum also developed cracks at the seats, and tubes continued to crack off in spite of attempts to keep the hydroxide concentration in the boiler water very low. Since the feedwater contained naturally considerable sulfate and chloride, the maintenance of low alkalinity resulted in high ratios of these salts to the total alkalinity. During the last nine months of operation the reported analyses show the sodium sulfate to total alkalinity ratio varying from 2.9 to above 20, and the sodium chloride to total alkalinity ratio varying from 2.3 to above 10. Even these ratios did not prevent the continual appearance of new cracks in the drums at the tube seats, and re-

TABLE II.—EFFECT OF SODIUM SULFATE AND SODIUM CHLORIDE ON THE FAILURE OF CONCENTRIC GROUND SPECIMENS.

Specimens of boiler flange steel.

Specimen	Stress, lb. per sq. in.	Grams per 100 g. of H_2O				Failure, hr.	Test Temperature, deg. Cent.	Pressure, lb. per sq. in.
		NaOH	Na_2SiO_3	NaCl	Na_2SO_4			
No. 62.114	60 000	25	0.32	25	75[a]	17	250	560
No. 62.117	60 000	25	0.8	25	75[a]	31	250	560
No. 62.126	50 000	40	1.0	40[a]	80[a]	84	225	350
No. 62.127	50 000	40	2.0	40[a]	80[a]	79	225	350
No. 62.124	50 000	40	0.5	40[a]	80[a]	95	200	215
No. 62.125	50 000	40	1.0	40[a]	80[a]	238	200	215

[a] Indicates amount added. This exceeds solubility.

[10a] Since this discussion was written these tests have been repeated with specimens of hot-rolled boiler flange steel. Sodium sulfate and sodium chloride even in very high concentrations did not prevent rapid cracking of the steel.

placed tubes frequently cracked within two or three weeks after installation. Microscopic examination showed the cracks to be intercrystalline. The experience with this boiler offers little support for the use of sodium sulfate or sodium sulfate and sodium chloride for the prevention of embrittlement.

The essential difference in the testing carried out by Messrs. Straub and Bradbury and that carried out by the writers arises from different viewpoints concerning the mechanism of concentration, and perhaps from certain differences in experimental details. Regarding the latter, it may be noted that Straub and Bradbury used hot-rolled steel while the writers used cold rolled. It would not appear that this should create a fundamental difference since the stress applied to hot-rolled steel during the test seems sufficient to create appreciable cold work in the metal. In any event it would seem desirable to use protective methods that were satisfactory in the presence of cold-worked metal for intercrystalline cracks are usually found in such areas in the boiler metal. It is apparent, however, that change in viewpoint concerning the first assumption about the mechanism of concentration, coupled perhaps with differences in experimental technique, have led to diametrically opposing results concerning the action of sodium sulfate and sodium chloride. The writers have not yet carried out experimental tests to determine the action of sodium aluminate at the higher pressures.

The statement in the paper, "The results of the tests conducted indicate that the method of testing boiler waters appears to give results directly applicable to boiler water treatment," now seems entirely unjustified. In the past the literal adherence to results of one type of testing and the application to boiler operation has led to serious errors. Perhaps the best example of one of these errors is the fact that previous experiments have shown that sodium sulfate would adequately prevent embrittlement, whereas, the experiments in this new testing unit show that this is not the case.

The writers would like to congratulate Messrs. Straub and Bradbury on the development of their new testing unit and to suggest only that the results be carefully interpreted and examined in comparison with all the other tests which are available for studying the embrittling action of a boiler water before they are applied in actual operation.

MR. F. N. SPELLER.[11]—Messrs. Schroeder and Straub are to be congratulated and should not be discouraged by apparent inconsistencies in test data. Everyone knows now that corrosion is a complicated series of problems; it isn't just one problem.

There is one question I should like to ask as to the form of the R_2O_3. Is it in solution, or is it in colloidal form, so to speak? The protection afforded by certain colloidal material against corrosion fatigue is proved in the case of oil well drilling mud. This type of protection may find a useful application in boiler water.

MR. C. H. FELLOWS.[12]—I should like to take this opportunity of expressing my thought that at the present time the situation surrounding the study of embrittlement is, perhaps, in the most healthy condition that it has ever been since we first became aware of the phenomenon of caustic embrittlement. The industry has been working on this problem and at the present time there are two outstanding investigators working on the subject. These two investigators have contributed a great deal to the promotion of knowledge of the subject, and like Mr. Speller I should like to voice my thought that we do not become discouraged and lessen our energies in this effort because there appear to be insurmountable obstacles and contradictions evident by the paper and by the discussion. We shall have ultimately a far better understanding of what caustic embrittlement is, and as a result, how we shall be able to prevent it most effectively.

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[12] Head, Chemical Division, Research Dept., The Detroit Edison Co., Detroit, Mich.

MESSRS. FREDERICK G. STRAUB[13] AND T. A. BRADBURY[13] (*authors' closure by letter*).—Mr. Partridge states that the experimental findings do not support the A.S.M.E. embrittlement ratios. Our interpretation is that the results of our research substantiate the A.S.M.E. ratios.

The operating data presented by Mr. Romer appear to correlate very closely with the laboratory data presented. There is no apparent effect of the R_2O_3 on prevention of embrittlement in the cases he cites due to the fact that this effect is not noticed until the higher operating pressures are reached.

It is rather difficult to discuss the results of the research reported by Messrs. Schroeder and Berk, since they do not give complete data nor a description of their testing units. Their conclusion reached from the history of one boiler, which had cracking occur within two or three weeks, is to the effect that the A.S.M.E. ratio is not effective. They forgot that there are available case histories of hundreds of boilers in which cracking has been stopped by maintenance of the A.S.M.E. ratio. We would be inclined to question workmanship more than the effectiveness of the A.S.M.E. ratio in the particular boiler they discuss.

The operators of power boilers are primarily interested in knowing whether their boilers are protected from embrittlement. If a laboratory test is developed, which in turn is correlated with actual operating data, this test might be of some service to the operators. However, if a test is developed, that has no correlation with operating data, we cannot see that it is necessary to abandon operating procedure which has been based upon years of experience in favor of the results of the uncorrelated laboratory tests.

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[13] Research Associate Professor, Chemical Engineering, and Laboratory Assistant, respectively, University of Illinois, Urbana, Ill.

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REMOVAL OF DISSOLVED SALTS FROM WATER BY EXCHANGE FILTERS

By P. G. Bird[1]

Synopsis

This paper discusses the newer organic types of exchanger bodies as applied to water treatment for the complete or partial removal of dissolved salts.

The methods of using these exchanger bodies is described for effecting various types of exchange with the results which should be expected from these materials.

The cation exchanger body may be used to replace zeolites and are advantageous in that respect for waters which cause disintegration of zeolites. The cation exchanger may also be regenerated with an acid and operates in the acid cycle. After regeneration it is possible when operating in conjunction with a zeolite softener to effect partial reduction of the dissolved solids equal to the carbonates or bicarbonates present in the water. The acid regeneration also makes it possible when operating in conjunction with an anion exchanger bed to pass the water through the beds in series to effect practically complete elimination of dissolved solids, except silica.

The removal of dissolved salts from solution, including sodium salts by means of exchange filters is a relatively new practice. As there are only a few such plants in operation, relatively little is known about the process, except by those who have had these units under observation.

To effect practically complete removal of dissolved salts from water, the water is passed through two filter beds arranged in series. The first bed, known as the "cation exchanger bed," removes the positive ions, or cations such as those of calcium, magnesium, and sodium. The second bed removes the negative ions, or anions, such as the chlorides, sulfates and nitrates and is referred to as an "anion exchanger bed."

The carbonate ions, or CO_2, which enter the system as bound CO_2 in the carbonates and bicarbonates of the raw water are not removed in either bed, but are released by aeration after the water has passed through the system.

For convenience, the exchangers used in this process are placed in two classifications.

Cation Exchange Bodies:

The first class of exchange body exhibits the property of cation exchange. The zeolites, which are characterized by being siliceous in nature, constitute one type of this class. There are several kinds of zeolites in commercial use, including both processed natural zeolites and synthetic zeolites.

Another type of cation exchange body

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is characterized by being organic and non-siliceous, rather than inorganic and siliceous. These non-siliceous organic exchange bodies may be further classified according to the nature of the raw materials from which they are derived and the method of manufacture employed in producing them.

Borrowman[2] was one of the first to employ the non-siliceous exchange bodies for water treatment. He used lignite coal as a substitute for zeolites. The materials described by Borrowman were regenerated with salt and operated in the sodium cycle. He pointed out that the activity of lignite was probably due to the humic acid and related acids present in lignite. As humic acid has organic carboxyl groups, the lignites may be classed as a carboxyl cation exchange body.

Adams and Holmes[3] and others described cation exchange bodies made by condensing phenolic compounds with an aldehyde and acid. Akeroyd and Broughton[4] believe that the activity of these exchangers is due to the presence of phenolic hydroxyl groups, and that the exchange of cations takes place on these groups. The phenolic cation exchangers may be regenerated with an acid, or in other words, hydrogen ions may enter into the exchange without destruction of the exchange body. This has not been practical with the siliceous cation exchangers or zeolites.

Another type of organic exchange body is characterized by the sulfonic groups. Burrell[5] states that these bodies may be produced by sulfonating lignins, wood, coals and similar substances by treatment with sulfuric acid. He has also described a method of testing these materials. The exchange capacity of organic substances may be increased by sulfonation, or may be imparted to bodies not previously having appreciable exchange capacity by sulfonation. This group is referred to as the sulfonic type of cation exchange body.

In these three general groups of cation exchangers (carboxyl, phenolic hydroxyl, and sulfonic) products ranging from high capacity exchangers having good physical properties to practically worthless materials may be found.

Cation Exchangers as Zeolites:

Many of these organic exchange bodies may be used in place of zeolites to soften water when salt is used for regeneration. These products are advantageous when used for treating water which is known to, or is likely to, cause destruction of siliceous zeolites.

As the industrial use of such products is relatively new, standards of exchange capacity, physical properties, and other characteristics have not been set. When used as zeolites, they should at least meet certain standards met by zeolites, unless other considerations are more important.

Zeolites require approximately ½ lb. of salt for regeneration for each 1000 grains of hardness removed from the water. Many zeolite installations require somewhat more salt than this, and some successfully use less. Organic exchangers should meet this standard of salt consumption when used as zeolites.

Wash water requirements vary considerably with the kinds of zeolite used and the method of operation, but ordinarily should not exceed 35 gal. per cu.

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[2] G. Borrowman, "Water-Purifying Material and Method of Preparing and Using the Same," U. S. Patent No. 1,793,670 (1931).

[3] B. A. Adams and E. J. Holmes, "Adsorptive Properties of Synthetic Resins," *Journal*, Soc. Chemical Industry, Vol. 54, No. 1, p. 6T (1935).

[4] E. I. Akeroyd and G. J. Broughton, "Alkali Adsorption by Synthetic Resins," *Journal of Physical Chemistry*, Vol. 42, p. 343 (1938).

[5] H. Burrell, "Organolites—Organic Base-Exchange Materials," *Industrial and Engineering Chemistry*, Vol. 30, p. 358 (1938).

ft. of exchanger bed for organic cation exchangers.

These organic substances may not be treated as zeolites in all respects. They are generally lighter in weight than zeolites and consequently require more freeboard with upflow than zeolites require. Some of these materials are so light that a 75 per cent bed expansion is experienced at an upflow rate of 7 gal. per sq. ft. per min., as compared to a 13.5 per cent bed expansion for a synthetic zeolite graded on the same screens. Designers must necessarily take this into consideration.

Whereas siliceous zeolites may impart silica to water, some of these organic exchange bodies will impart soluble organic matter, as well as tastes and odors to water. This disadvantage has been recognized[6] and efforts to stabilize such products have been made.

Acid Regeneration:

The phenolic hydroxyl and sulfonic cation exchangers may be regenerated with acids. Sulfuric acid is generally preferred because of its lower cost. When these bodies are regenerated with acid, and water containing dissolved salts is passed through a bed of such material, the positive ions of the salts are exchanged for hydrogen ions and are retained in the exchanger bed. The result is that the effluent is free of sodium, magnesium, calcium, and other positive ions, except hydrogen. The effluent bears acids rather than salts. The carbonates, chlorides, sulfates, and nitrates in the raw water are converted to carbonic, hydrochloric, sulfuric and nitric acids, respectively, and these acids are present in the effluent in amounts chemically equivalent to the salts in the raw water. When the exchange body is exhausted, the amount of acids in the effluent will decrease, and this decrease will be accompanied by a corresponding increase in salts. The first salts to appear will be sodium salts with calcium and magnesium salts being retained. An analysis of the effluent will show that the sodium salts may exceed the sodium salts in the raw water, indicating that after the exchanger bed is exhausted as a hydrogen exchanger it will function for some time as a zeolite, and that the sodium absorbed while acting as a hydrogen exchanger has put the bed into condition to act as a zeolite.

Acid Effluent Neutralization:

The acid effluent from the cation exchanger is further treated according to the use to which it is to be put. If desired, the mineral acidity (hydrochloric, sulfuric and nitric acids) may be neutralized by suitable alkalies, raw alkaline water, or zeolited bicarbonate water, and the carbonic acid removed by aeration. The net result of such a process is to reduce the carbonates and bicarbonates or alkalinity of the raw water. Such a process results in only partial reduction of dissolved solids as it does not reduce the chlorides, sulfates, nitrates or silica content and may leave the water acidic, unless careful control is exercised.

Anion Exchangers:

Instead of neutralizing the acid effluent it may be sent to a second exchanger bed which will remove the acids. In this class of exchanger the negative, rather than the positive, ions enter into the exchanger process. This class of exchange body was described by Adams and Holmes[3] in 1934. Those products were resins derived by condensing aromatic amines with an aldehyde and an acid.

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[6] H. L. Tiger and P. C. Goetz, "Stabilizing Base Exchanging Humates," U. S. Patent No. 2,069,564.

Other methods of preparing anion exchange bodies, other than by condensation have been suggested, such as halogenating a suitable body and following this by amination.

The anion exchange bodies examined appear to have very little capacity for carbonic acid, a good capacity for hydrochloric acid, and a still higher capacity for sulfuric acid. A high-grade anion exchange body will have a capacity greater than 6000 grains per cu. ft. for hydrochloric acid, and greater than 15,000 grains per cu. ft. for sulfuric acid.

Removal of Acids:

The entire process of removing all dissolved salts from water consists of passing water through a bed of cation exchange bodies to remove the positive metal ions, and then through a bed of anion exchange bodies to remove the acid or negative ions. The effluent from this two-stage process will contain carbon dioxide equal to the bicarbonates in the raw water, which gas is later liberated by aeration.

The effluent will be free of sulfates, calcium, and magnesium and will be substantially neutral if good liberation of carbon dioxide is obtained. The residual carbon dioxide should not be over 15 p.p.m. An atmospheric type of aerator will leave the water saturated with oxygen with the result that the treated water is as corrosive as distilled water. The water will contain from a trace to a few parts per million of sodium bicarbonate, sometimes a trace of chlorides, and silica equal to that in the raw water. There may or may not be a reduction in organic matter. When insoluble exchange bodies are used, some organic matter may be removed from the water, the maximum reduction observed in soluble organic matter being from 30 to 6 p.p.m. If the organic exchangers are not completely stable, an increase in organic matter may result.

Equipment:

As the cation exchanger is regenerated with an acid, it operates in an acid cycle. This necessitates acid-proof construction if long equipment life is desired. A very satisfactory material of construction is rubber-lined steel. Wood tanks have been used with success for low-pressure work, and hard rubber has been utilized for very small units. If traces of copper and other heavy metals in the water are not objectionable, copper piping and brass valves may be used, but where these metals are objectionable, rubber-lined steel pipe and rubber-lined diaphragm valves are best.

The tanks and fittings used for the anion exchanger must also be resistant to the action of acids, as acidic water from the first stage enters the second stage. The anion exchange body is regenerated with an alkali and operates on an alkaline cycle during regeneration. This requires materials of construction that withstand alkalies as well as acids.

Efficiency:

In calculating chemical efficiency it is convenient to convert all salts, acids and caustic to terms of calcium carbonate. By doing this all chemicals involved are put on a common basis. For example, a water having an analysis on the "as is" basis would be converted to terms of calcium carbonate as shown below:

	HYPOTHETICAL COMBINATIONS, GRAINS PER GAL.	
	"AS IS" BASIS	AS CaCO$_3$
Calcium carbonate	5.0	5.0
Magnesium carbonate	4.0	4.8
Calcium sulfate	6.0	4.4
Sodium sulfate	7.0	4.9
Sodium chloride	3.0	2.6
Total	25.0	21.7

The total in terms of calcium carbonate constitutes the dissolved solids to be removed and represents the load on the cation exchanger bed. The cation exchanger bed should have a total capacity of 21,700 grains to remove the positive metal ions from 1000 gal. of such water. The anion exchanger bed would not have as much exchange capacity as the first bed, since it would be required to remove only the chloride and sulfate ions. For 1000 gal. of this water the anion exchanger bed should have a capacity of only 11,900 grains, as the carbonates are not removed in the second bed.

The acid required for regeneration, based on 66° Baumé sulfuric acid, will vary from 0.2 to 0.4 lb. of acid per 1000 grains of dissolved solids removed by the cation exchanger bed. The average acid consumption will be from 0.25 to 0.35 lb. per 1000 grains in terms of calcium carbonate.

TABLE I.

Efficiency, per cent	Acid[a] per 1000 grains (as $CaCO_3$), lb.	NaOH per 1000 grains of Acid (as $CaCO_3$), lb.	Salt per 1000 grains of Hardness (as $CaCO_3$), lb.
100......	0.150	0.114	0.167
80......	0.188	0.143	0.209
60......	0.250	0.191	0.278
40......	0.375	0.286	0.417
20......	0.750	0.572	0.835

[a] 66° Baumé sulfuric acid.

The caustic required is based on the sum of the chlorides, sulfates and other negative ions removed by the anion exchanger. Very efficient regeneration of the anion exchangers is obtained when caustic soda is used as the regenerent. Chemical efficiencies up to 80 per cent have been obtained, and a chemical efficiency based on chemical equivalents of 70 to 75 per cent can be expected for normal operation.

A comparison of chemical efficiency of acid and caustic soda regenerations for the removal of dissolved salts, together with a comparison of salt efficiency for zeolite regeneration, is interesting in that at the rate of 0.5 lb. of salt per 1000 grains of hardness removed in zeolite softening the chemical efficiency is only 33.4 per cent when based on chemical equivalents (Table I).

Control:

The strength of acid solution used for regeneration will depend to some extent on the cost of the water. Acid dilutions varying from 0.5 to 4.0 per cent have been used. Somewhat greater efficiency is obtained with the more dilute acid but the amount of water required for regeneration is increased due to the high dilution.

It is convenient to pour the concentrated acid into the water in an acid vat and obtain an initial dilution of 10 to 15 per cent acid. There is some heat generated, but not enough to cause sputtering. The final dilution is accomplished by means of an injector.

The caustic solution strength may be carried over a wide range, but the most efficient solution to use is very dilute. Less than 1 per cent solutions of caustic soda are satisfactory.

The control of this process is simple. The effluent from the cation exchanger bed will be acidic to methyl orange, and this acidity is chemically equivalent to the sulfates, chlorides, and nitrates in the raw water. When tests of the effluent show that the acidity of the effluent has decreased below this amount, the cation exchanger bed should be regenerated.

Tests of the effluent from the anion exchanger are also simple. The effluent from the second stage is neutral or slightly alkaline to methyl orange in normal operation. Whenever a test with methyl orange shows a slight acidity the anion exchanger bed should be regenerated. These same tests will indicate when the two beds are ready to be put in operation after regeneration. Automatic control devices have been worked out to warn the operator when either bed should be regenerated. These control devices are valuable supplemental adjuncts to chemical control.

The time required for regeneration will vary from 30 to 60 min. for the cation exchanger, and from 45 to 90 min. for an anion exchanger. Both beds may be regenerated at the same time with a total time consumption of not over ninety minutes.

With either cation or anion exchangers, flow rates up to 6 and 7 gal. per min. per sq. ft. may be used without loss of exchange capacity with beds 30 in. or more in depth. Lower flow rates than 1 gal. per min. per sq. ft. on anion exchanger beds is sometimes accompanied by a slight increase in sodium bicarbonate content in the effluent. It is difficult to wash all of the caustic used in regeneration out of the anion exchanger bed with the result that small quantities are leached out during operation. This is the source of the sodium bicarbonate in the effluent, and if the flow rate is very low the concentration of sodium bicarbonate will increase over the 2 to 5 p.p.m. present at flow rates from 3 to 6 gal. per min. per sq. ft.

More wash water is required to wash out the excess caustic from the anion exchanger bed after regeneration than is required to wash out the excess acid from the cation exchanger bed. The over-all water requirements for regeneration and washing for both beds will vary between 30 and 40 gal. per cu. ft. of exchange bodies in the system depending upon the dilutions of acid and caustic soda used.

Conclusions:

These newer exchange bodies have increased the usefulness and scope of exchange processes. By the proper combination of such materials, a great improvement may be obtained over the zeolite method of treating water, particularly for boiler feedwater purposes.

It appears that these exchange bodies will be used for treating water to a greater extent than has been the case with zeolites. However, these exchange processes should not be looked upon as a "cure all" for water treatment problems. If the water is to be used for boiler feed, chemical aftertreatment and control will be required for corrosion and silica-scale prevention. The choice of treatment will necessarily be made on the basis of cost and the operating conditions to be met. These factors can be determined by a careful study of the recent advances in both the chemical and exchange process of water treatment.

DISCUSSION

Mr. J. D. Yoder[1] (*presented in written form*).—The paper by Bird gives a clear presentation of the functional properties of the new class of ion exchange substances. They have enlarged the significance of the term "zeolite" and have increased the scope of the application of zeolite in the industries. The characteristics that he ascribed to these materials are quite in accord with the findings of independent investigators.

The advantage of hydrogen zeolite when supplemented with sodium zeolite as described in this paper, was promptly recognized by engineers who have made a special study of boiler feedwater treatment. For a number of years engineers have been seeking methods of treatment which remove hardness from the water, reduce the alkalinity to as low a figure as possible and likewise reduce the total solids. Heretofore lime treatment was the only practical method employed for removing both hardness and alkalinity, but this treatment did not remove the calcium and magnesium below their points of solubility which ranged from approximately 20 to 40 p.p.m. in terms of calcium carbonate, depending upon the temperature and excess of sodium carbonate alkalinity. Former zeolites reduced the hardness to substantially zero but they made no reduction in alkalinity. The hydrogen zeolite used in conjunction with a sodium zeolite softener at once removes all of the hardness and gives a water of zero alkalinity if desired.

Low alkalinity of boiler feedwater offers a number of advantages. It reduces total solids, particularly when sodium sulfate must be added to maintain desired sulfate to carbonate ratios; it gives more favorable steaming conditions and it reduces the free CO_2 in the steam. The latter is of special value when steam is used for heating because the higher the CO_2 in the steam the lower will be the pH value of the condensate and the greater the corrosion of condensate lines. Public utilities recognize that the less CO_2 they deliver to their steam heating mains, the fewer will be the complaints of corrosion from their customers.

To obtain these advantages, plus substantially no hardness in the feedwater have been the determining causes for the selection of this method of treatment of boiler feedwater for boiler pressures as high as 1200 lb. and in capacities as large as 50,000 gal. per hr.

Bird calls attention to these products being non-siliceous. Accordingly, this type of zeolite may be used for softening water after the silica has been removed by suitable equipment. For this service the zeolite is generally operated on the sodium cycle. This type of zeolite has already become the preferred method of softening water from which slica has been removed.

It is therefore clear that the first step described by Bird for the removal of mineral salts is well established in practice.

The utility of the second step has not yet been established by extensive usage although there are a number of relatively small plants in operation. The indications are quite favorable for the early application of demineralizing processes for the treatment of water.

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THE ESTIMATION OF SODIUM IN WATER SUPPLIES BY AN INDIRECT METHOD

By J. B. Romer,[1] W. W. Cerna[1] and H. F. Hannum[1]

Synopsis

A method for the estimation of sodium is presented which has been in use for a number of years in our laboratory. This method, while indirect, nevertheless gives good results on most industrial waters.

The method is based on the principle that all inorganic basic ions present in the solids obtained on evaporating the water sample to dryness are converted, by controlled fuming with sulfuric acid, to either sulfates or oxides.

With the exception of sodium, potassium and lithium, all of the anions present after this treatment can be readily and accurately determined by standard quantitative methods.

With the above data available it is possible to estimate, by simple calculations, the amount of sodium present.

A series of samples was prepared by dissolving in distilled water various amounts of those compounds common to industrial waters. These samples were analyzed and the results show that sodium is quite accurately determined by this method.

The estimation of sodium in water by the indirect method has been in use in our laboratory for a number of years. The basis of the method is found in Low's "Technical Methods of Ore Analysis."[2]

During the years in which this method has been used we have found the results to be very reliable as is borne out by the check work being reported at this time. The body of this paper covers the analytical procedure and the results of the analyses of a number of water samples of known composition.

For those analysts who evaporate their water samples to dryness and determine certain constituents by standard methods, only a few modifications are required in order to obtain the sodium content by this method. Excellent results are readily obtained, and its presentation at this time may be of interest to others engaged in the chemical analysis of water.

Analytical Procedure:

It has been found that about 0.1 g. of residue on evaporation makes a very convenient quantity of solids for use in this test. Therefore, sufficient water to produce approximately this quantity of solids is transferred by means of an accurately calibrated pipette to a weighed platinum dish and evaporated to dryness at a temperature just below ebullition. The evaporating bath must be protected from foreign matter such as dust, fumes, etc. If desired, this step can also be used for the determination of the total solid residue obtained on evaporation, in which case the dish and residue are placed in an oven at 105 C., dried for two hours, cooled and weighed.

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[1] Chief Chemist, Assistant Chemist, and Analyst, respectively, The Babcock & Wilcox Co., Barberton, Ohio.

[2] A. H. Low, "Technical Methods of Ore Analysis," Sixth Edition, John Wiley and Sons, New York City (1913).

Sulfated Residue:

To the dish containing the residue after evaporation, concentrated sulfuric acid, 3 to 5 ml., is gradually added and then the dish is slowly rotated and tilted so that the acid comes in contact with every portion of the residue. The dish is then placed upon a hot plate or over a very low flame and the excess sulfuric acid is driven off by fuming. The heating should not be conducted too rapidly, as this may cause spattering or mechanical loss of the residue. The dish is then heated very carefully to redness, so as to destroy any organic matter, and after cooling, is re-treated with 3 ml. of concentrated sulfuric acid as above, and again heated in the same manner as before, to drive off the excess acid.

The dish is now placed in a desiccator, allowed to cool, and then weighed. The difference between the weight of the clean dish and the dish plus the sulfated residue is recorded as the weight of the sulfated residue.

After the above treatment, all sodium, potassium, calcium, and magnesium present will be in the sulfate form, all iron and aluminum will be in the oxide form, and the organic matter will have been eliminated. The second treatment of the residue with sulfuric acid can frequently be dispensed with, since it is necessary only in cases where the quantity of organic matter is very high or when considerable residue is being sulfated. If desired, this sulfated residue can be used for the determination of silica, iron and aluminum oxides, calcium, and magnesium in the order named, by the usual standard procedures briefly outlined as follows:

Digest the sulfated residue with strong hydrochloric acid for 15 min. on a hot plate, dilute with water, filter, wash with dilute HCl and water, etc. Weigh residue as SiO_2.

Iron oxide and alumina are determined in the filtrate by making the solution just alkaline to methyl-red indicator, boiling, filtering, etc.

Calcium is determined in the filtrate by adding excess ammonium hydroxide, heating to boiling, adding ammonium oxalate, boiling a few minutes, settling, filtering, etc.

Magnesium is determined by neutralizing the filtrate from the calcium separation with hydrochloric acid, adding ammonium phosphate followed by a 5 to 10 per cent excess of ammonia, stirring, allowing to settle, filtering, etc.

If preferred, these determinations can be carried out on separate portions of water by other methods. Any method so used, however, should be accurate, as *any inaccuracy in the results will directly affect the accuracy of the determined sodium.*

Calculation of Sodium Concentration:

Calculate all calcium and magnesium found to their respective sulfates. The sum of the calcium sulfate, magnesium sulfate, iron and aluminum oxides, and silica is subtracted from the weight of sulfated residue. The difference in weight represents the total sodium as sodium sulfate. This weight is then calculated to sodium, the result in milligrams equivalent sodium obtained per liter of water used for the determination represents parts per million of sodium in the sample.

Sample Calculation:

Typical data as would be obtained in connection with the analysis of a sample of water by the preceding method and the estimation of the total sodium from these data are given below:

Size of sample			100 ml.
Weight of sulfated residue, g			0.1000
Silica	SiO_2	0.0025	
Iron and aluminum oxides	R_2O_3	0.0010	
Calcium, Ca = 0.0050: equivalent to	$CaSO_4$	0.0170	
Magnesium, Mg = 0.0020: equivalent to	$MgSO_4$	0.0099	
Total weight, g		0.0304	0.0304
Total sodium as sodium sulfate, g.	Na_2SO_4		0.0696
Equivalent sodium, g	Na		0.0215
Sodium, Na, p.p.m		215	

Experimental Data:

To demonstrate the reliability of this method of determining sodium, a series of water samples was prepared by adding weighed amounts of pure chemicals to distilled water. These samples were then analyzed by the method just described.

Calcium and magnesium bicarbonate solutions were prepared by passing carbon dioxide through a suspension of carbonates. The calcium sulfate and magnesium chloride were added as hydrated salts and computed to the anhydrous form. Organic matter was added in the form of pure tannic acid.

A total of twelve samples was prepared. They contained various combinations of the constituents normal to raw and boiler waters. These can be divided into three series. The approximate amount, in parts per million, of the major constituents present is given below:

	SERIES A	SERIES B	SERIES C
Calcium and magnesium as $CaCO_3$, p.p.m.	25 to 200	30 to 215	none
Sodium alkalinity as Na_2CO_3, p.p.m.	none	5 to 65	250 to 300
Total sodium as Na, p.p.m.	10 to 80	10 to 350	370 to 390
Total solids	250 to 350	275 to 1000	1000
Phosphate as PO_4, p.p.m	none	none	0 to 67
Sodium nitrate as $NaNO_3$, p.p.m.	none	none	0 to 100

The complete record of the compounds present in these samples, as well as the

TABLE I.—SERIES A, NO SODIUM ALKALINITY PRESENT.

	TEST No. 1	TEST No. 2	TEST No. 3	TEST No. 4
Compounds Present, p.p.m.:				
Sodium sulfate, Na_2SO_4	120	15	...	...
Sodium chloride, NaCl	80	10	200	25
Calcium bicarbonate, $Ca(HCO_3)_2$	16	130	...	...
Calcium sulfate, $CaSO_4$	4	32	8	95
Magnesium bicarbonate, $Mg(HCO_3)_2$	17	139	17	139
Magnesium chloride, $MgCl_2$	...	...	6	15
Organic matter	25	25	25	25
Total solids	262	351	256	299
Total Basic Ions Added and Recovered, p.p.m.:				
Sodium, added	70.3	8.8	78.7	9.8
Sodium, recovered	67.5	6.5	76.5	5.3
Sodium, difference	−2.8	−2.3	−2.2	−4.5
Calcium, added	5.2	41.3	2.3	27.9
Calcium, recovered	5.0	41.8	2.5	27.8
Magnesium, added	2.9	23.1	4.4	26.9
Magnesium, recovered	3.2	21.0	4.4	24.3

TABLE II.—SERIES B, SODIUM ALKALINITY (LOW CONCENTRATION) PRESENT.

	TEST No. 5	TEST No. 6	TEST No. 7	TEST No. 8
Compounds Present, p.p.m.:				
Sodium hydroxide, NaOH	...	...	...	50
Sodium carbonate, Na_2CO_3	...	...	50	...
Sodium bicarbonate, $NaHCO_3$	10	80	...	...
Sodium sulfate, Na_2SO_4	10	80	550	550
Sodium chloride, NaCl	5	40	375	375
Calcium bicarbonate, $Ca(HCO_3)_2$	194	32	...	...
Magnesium bicarbonate, $Mg(HCO_3)_2$	139	17	...	...
Organic matter	25	25	25	25
Total solids	383	274	1000	1000
Total Basic Ions Added and Recovered, p.p.m.:				
Sodium, added	7.9	63.5	347.3	354.3
Sodium, recovered	4.5	60.5	346.5	354.2
Sodium, difference	−3.4	−3.0	−0.8	−0.1
Calcium, added	48.0	8.0	.....	.....
Calcium, recovered	47.5	8.0	.....	.....
Magnesium, added	23.1	2.9	.....	.....
Magnesium, recovered	20.6	3.2	.....	.....

TABLE III.—SERIES C, SODIUM ALKALINITY (HIGH CONCENTRATION) PRESENT.

	TEST No. 9	TEST No. 10	TEST No. 11	TEST No. 12
Compounds Present, p.p.m.:				
Sodium hydroxide, NaOH	150	150	150	250
Sodium carbonate, Na_2CO_3	50	50	50	...
Sodium phosphate, Na_2HPO_4	...	...	100	100
Sodium nitrate, $NaNO_3$	...	100	...	...
Sodium sulfate, Na_2SO_4	475	450	450	425
Sodium chloride, NaCl	300	225	225	200
Organic matter	25	25	25	25
Total solids	1000	1000	1000	1000
Total Basic Ions Added and Recovered, p.p.m.:				
Sodium, added	379.2	368.7	374.0	391.4
Sodium, recovered	377.2	366.9	370.1	383.1
Sodium, difference	−2.0	−1.8	−3.9	−8.3

results of each analysis, is given in Tables I, II and III.

The pertinent data as to the amounts of calcium, magnesium, phosphate and sodium added and the recovery of sodium have been assembled in Table IV.

Table IV brings out the fact that, under most conditions, the recovery of the sodium was well within analytical limits. It is to be noted however, that when the magnesium content is high there is a tendency towards low recovery of the sodium. This is only of moment when the sodium content is low. The cause of this condition lies in the fact that magnesium sulfate when heated tends to decompose with the formation of magnesium oxide and consequential lowering of the weight of the sulfated residue.

TABLE IV.—DATA ON SODIUM RECOVERY.

Sample	Sodium, p.p.m.			Magnesium Added, p.p.m.	Calcium Added, p.p.m.	PO_4 Added, p.p.m.
	Added	Recovered	Lost			
A-4....	9.8	5.3	4.5	26.8	27.9	None
B-5....	7.9	4.5	3.4	23.1	48.0	"
A-2....	8.8	6.5	2.3	22.8	41.3	"
A-3....	78.7	76.5	2.2	4.4	2.3	"
B-6....	63.5	60.5	3.0	2.9	8.0	"
A-1....	70.3	67.5	2.8	2.9	5.2	"
B-8....	354.3	354.2	0.1	None	None	"
B-7....	347.3	346.5	0.8	"	"	"
C-10...	365.7	366.9	1.8	"	"	"
C-9....	379.2	377.2	2.0	"	"	"
C-11..	374.0	370.1	3.9	"	"	67.0
C-12...	391.4	383.1	8.3	"	"	67.0

Phosphates sometimes produce erratic results as is to be noted in samples C-11 and C-12. These two samples are quite similar in their principal features yet the recovery of sodium is much better in sample C-11 than in C-12.

It must be realized that this method throws all errors into the sodium determination. These samples were prepared from the highest purity chemicals available and the C series samples contained considerable caustic soda which as we all known is hard to handle with absolute accuracy. This condition is reflected in sample C-12.

Potassium unless determined by gravimetric methods is the one element that interferes with this method so as to produce appreciable error. It will be present in the sulfated residue as potassium sulfate but will be calculated as if it were sodium sulfate thus producing an error proportionate to the amount present and the relation of the molecular weights. This condition can be compensated, whenever one desires, by determining the amount of potassium and allowing for it in the sulfated residue by subtracting the equivalent potassium sulfate weight from the weight of the sulfated residue before calculating the equivalent sodium.

Conclusion:

In conclusion the authors wish to state that the data submitted as a part of this paper bear out their observations made while analyzing many samples of water that this method, when carefully handled, permits the estimation of the sodium content of waters with a high degree of accuracy. The simplicity of the method increases its usefulness and recommends it in place of more tedious methods.

DISCUSSION

Mr. Robert T. Sheen[1] (*presented in written form*).—The method described in the paper has been used in our laboratories with some modification. When it is desirable to determine sodium, an analysis is also usually required for both total solids and organic matter and the total solids are determined by the evaporation to dryness in the manner described. To the dish containing the residue following the evaporation, we add 3 to 5 ml. of hydrochloric acid, allowing the acid to come into contact with each portion of residue and evaporate the acid just to dryness in an oven at 105 C., cool and weigh. The purpose of the hydrochloric acid is to remove any carbonate that may be present that would be driven off together with the organic matter. The dish with the sample is then placed on a hot plate or over a flame to burn off the organic matter, cooled and weighed and the difference between this weight and the weight following evaporation with the hydrochloric acid will give the organic matter present in the sample. The residue may then be sulfated in the manner described by the authors. Satisfactory results are obtained in this manner to include the several determinations of total solids, organic matter and sodium. The excellent results obtained by the authors definitely show this method to be dependable, and particularly can this be said to be true of boiler waters. It should possibly be considered as a method for the sum of sodium and potassium on raw or natural waters.

Mr. D. S. McKinney[2] (*presented in written form*).—I wish to propose a variation of the method described by the authors, to be used for samples containing phosphates. It will be noted that the greatest errors occur in samples C-11 and C-12 containing phosphates. This is probably due to three defects in the procedure.

1. All calcium, magnesium, and sodium are assumed to be present as sulfates in the sulfated residue. In waters containing phosphate this assumption is probably not correct, since at least a portion of the phosphate must remain after ignition.

2. Due to the indefinite time and temperature of the ignition one is uncertain as to what form of phosphate is present, that is, orthophosphate or pyrophosphate.

3. Failure to remove phosphate may cause calcium and magnesium to precipitate as phosphates with the R_2O_3 precipitate on addition of ammonium hydroxide. They would not then be included in the calculation as sulfates.

To obviate these defects, one may remove the phosphate as follows:

Evaporate the sample to about 50 ml. volume and add $FeCl_3$ solution of known strength sufficient to form $FePO_4$ with the phosphate present. In order to be sure of removal, a reasonable excess of $FeCl_3$ may be added. The sample is then made alkaline with ammonia and boiled until just acid to litmus. The precipitate of mixed $FePO_4$ and Fe

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TABLE I.—COMPARATIVE ANALYSES OF DUPLICATE WATER SAMPLES, BABCOCK & WILCOX CO. AND DUQUESNE LIGHT CO.

	River Water						Boiler Feedwater						Boiler Water					
	A		B		C		A		B		C		A		B		C	
	B & W	D L	B & W	D L	B & W	D L	B & W	D L	B & W	D L	B & W	D L	B & W	D L	B & W	D L	B & W	D L
ANALYSES, P.P.M.																		
Total solids	195.5	160.0	152.7	199.9	208.4	189.4	4.8	3.1	36.4	28.6	41.0	37.0	579.5	664.7	5570.2	5418.4	533.8	923.1
Dissolved solids	189.5	129.1	132.7	188.3	205.3	186.9	4.8	3.1		28.6	36.0	34.8	578.59	646.8		5417.0	527.8	920.8
Suspended solids	6.0	30.9	20.0	11.6	3.1	2.5	0	0	trace	0	5.0	2.2	0.91	17.9	trace	1.4	6.0	2.3
Alkalinity solids	23.0	8.8	0	6.8	40.0	29.3	0	0.5	7.7	4.0	7.1	8.9	27.7	26.4	1547.7	1668.0	147.4	124.2
Bicarbonate alkalinity		8.8		6.8		29.3		0.5		1.1		8.9		22.4		0		0
Carbonate alkalinity		0		0		0		0		2.9		0		4.0		213.0		34.8
Hydroxide alkalinity		0		0		0		0		0		0		0		1445.0		89.4
Free CO_2		4.3		3.4		6.0		1.3		0		1.0		0		0		0
Organic matter		2.6		0		6,6		0.1		0.4		0.6		0.2		38.0		0.8
Iron and aluminum oxides	0.7	1.2	0.3	1.7	1.4	1.0	trace	0.1	0.6	0.2	0.5	0.6	2.7	0.8	8.4	11.4	1.5	1.4
Silica	6.0	6.4	6.0	6.3	10.9	11.0	1.0	0.8	2.9	2.6	3.9	4.7	158.5	188.4	12.0	32.0	6.0	4.0
Calcium	18.8	19.8	21.1	23.5	32.8	33.7	0.1	0.1	0.2	3.7	4.0	4.6	3.6	5.9	4.0	4.0	35.9	39.5
Magnesium	5.5	4.9	4.8	5.9	11.7	7.4	trace	0.1	trace	0.1	0.7	0.8	trace	0.4	trace	1.6	trace	0.9
Sodium	*10.7*	*8.2*	*9.8*	*11.1*	*11.0*	*5.1*	*0.7*	*0.3*	*11.2*	*2.6*	*4.2*	*3.4*	*136.2*	*154.9*	*2275.4*	*2003.3*	*285.5*	*273.8*
Carbonate	9.3	0	0	0	24.8	0	0	0	2.9	1.7	1.3	0	5.5	2.4	90.5	127.9	39.5	20.9
Bicarbonate	9.1	10.7	0	8.3	10.5	35.8	0	0.7	0	1.4	6.1	10.8	22.5	29.3	0	0	0	0
Hydroxide	0	0	0	0	0	0	0	0	1.0	0	0	0	0	0	474.9	491.2	27.7	30.4
Sulfate	47.0	56.1	74.2	82.6	76.7	71.4	0.3	0.6	13.5	9.6	8.0	8.0	97.1	110.9	3038.4	2356.1	173.3	158.0
Chloride	15.2	14.3	12.1	9.9	16.9	15.1	1.0	trace	1.9	0.9	5.0	3.7	125.2	148.6	174.3	182.5	271.6	289.5
Nitrate		0		0.3		0.9		trace		trace		0		0		11.1		1.1
Phosphate						0						trace						trace
ANALYSES, MILLI-EQUIVALENTS																		
Cations:																		
Calcium	0.938	0.988	1.05	1.17	1.64	1.68	0.005	0.005	0.010	0.184	0.200	0.230	0.180	0.294	0.200	0.200	1.79	1.97
Magnesium	0.452	0.403	0.395	0.485	0.962	0.608		0.008		0.008	0.058	0.066		0.033		0.132		0.074
Sodium	0.465	0.357	0.426	0.483	0.479	0.222	0.030	0.013	0.487	0.113	0.183	0.148	5.92	6.74	98.98	87.14	12.42	11.91
Total	1.855	1.748	1.871	2.138	3.081	2.510	0.035	0.026	0.497	0.305	0.441	0.444	6.100	7.067	99.18	87.572	14.21	13.954
Anions:																		
Carbonate	0.310	0	0	0	0.826	0	0	0	0.097	0.057	0.043	0	0.183	0.080	3.01	4.26	1.32	0.696
Bicarbonate	0.149	0.175	0	0.136	0.172	0.587	0	0.011	0	0.023	0.100	0.177	0.369	0.481	0	0	0	0
Hydroxide	0	0	0	0	0	0	0	0	0.059	0	0	0	0	0	27.92	28.88	1.63	1.79
Sulfate	0.978	1.17	1.54	1.72	1.60	1.49	0.006	0.012	0.281	0.200	0.166	0.166	2.02	2.36	63.20	49.01	3.60	3.29
Chloride	0.429	0.403	0.341	0.279	0.477	0.426	0.028		0.054	0.025	0.141	0.104	3.53	4.19	4.92	5.15	7.66	8.16
Nitrate		0		0.005		0.014										0.179		0.018
Phosphate						0												
Total	1.866	1.748	1.881	2.140	3.075	2.517	0.034	0.023	0.491	0.305	0.450	0.447	6.102	7.111	99.15	87.479	14.21	13.954
Difference	0.011	0.000	0.010	0.002	0.006	0.007	0.001	0.003	0.006	0.000	0.009	0.003	0.002	0.044	0.03	0.093	0.00	0.000

$(OH)_3$ is removed by filtration, the filtrate returned to the platinum dish and the procedure as described in the paper is followed from this point onward. Iron may be separately determined if necessary. In alkaline waters it is usually so low as to be negligible.

MESSRS. MAX HECHT[3] AND S. F. WHIRL[4] (*presented in written form*).—The authors' method for the indirect determination of sodium is especially suited to the mass routine analyses of industrial waters. The suggested manipulative procedure is readily adaptable to any laboratory and the results obtained are reasonably accurate.

The authors point to the low recovery of sodium in the presence of phosphates and to similar low recovery when the magnesium content is high (Table IV). In attempting to substantiate the authors' observations, some experimental work has been initiated by the discussers and the preliminary data indicate that (*a*) the presence of phosphates tends to increase rather than decrease the weight of the sulfated residue over the theoretical, and (*b*) the recovery of magnesium sulfate is complete. Errors due to the presence of phosphates result from failure to volatilize them completely during the fuming process. The double procedure recommended by the authors is of particular significance if phosphates are present in appreciable amounts. Hillebrand[5] points out that the extent of volatilization of phosphates during fuming depends on the amount of sulfuric acid used and the rate at which it is driven off. Preliminary investigation indicates that high phosphates may necessitate even more than two sulfations. With reference to the authors' statement concerning the decomposition of magnesium sulfate, the International Critical Tables fail to show a decomposition temperature for this compound, but do give a melting point temperature of 1185 C., which appears to be considerably above the final fuming temperature, "redness," used by the authors.

In 1927 comparative analyses on duplicate samples were made by the laboratories of the Babcock & Wilcox Co. and the Duquesne Light Co., using the methods as outlined respectively in this paper and as given below. These results are tabulated in the accompanying Table I as parts per million and for further comparison, calculated in terms of milli-equivalents.

The authors advisedly point out that the results for sodium include all errors in the determination of constituents used in the calculation. With this consideration, the results obtained by the two laboratories on industrial waters compare favorably with the research data of the authors.

It is observed from the data in Table I that the balance of anion and cation equivalents for the individual laboratories is within the allowable errors suggested by Stabler.[6] The lack of agreement of the same order between the two laboratories may be due to differences in details of procedure; even here, however, cross balancing the anion and cation equivalents of the two laboratories gives results which in most cases fall within Stabler's allowable errors.

The discussers' data were secured by a somewhat more amplified procedure[7] than used by the authors. From a single filtered aliquot sample, the discussers secure data for dissolved solids, ignition loss (organic matter), and the other constituents mentioned by the authors. The steps of the procedure are as follows:

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[3] Advisor, Power Stations Chemistry, Pittsburgh, Pa.

[4] Chief Chemist, Duquesne Light Co., Pittsburgh, Pa.

[5] W. F. Hillebrand, "Analysis of Silicate and Carbonate Rocks," *Bulletin No. 700*, U. S. Geological Survey, p. 116 (1919).

[6] Herman Stabler, "Some Stream Waters of the Western United States," *Water Supply Paper 274*, U. S. Geological Survey, p. 11 (1911).

[7] The discussers' method was secured in 1920 in a private communication from the late M. F. Newman of the W. B. Scaife Co., who detailed a method used by their chief chemist, the late H. R. Jessel.

1. Evaporating, drying of residue at 105 C. and weighing.
2. Carbonating moistened residue with carbon dioxide, drying at 105 C. and weighing.
3. Ignition of residue at dull red heat, cooling, recarbonating with carbon dioxide, drying at 105 C., and weighing.
4. Treating residue with concentrated sulfuric acid, fuming at a temperature not exceeding 800 C. and weighing.
5. Chemical analysis of residue.

Evaporation in all steps of the procedure is made in the same platinum dish. Previous to each weighing, the dish is cooled in a desiccator. It is imperative that this detail be observed, particularly following sulfation, since high magnesium content residues absorb atmospheric moisture avidly. The carbonating steps are made to secure a reasonably accurate value for the organic matter and are used on waters containing either bicarbonates or hydroxides.

Some reference books suggest the use of sodium carbonate in steps 2 and 3. To avoid corrections for the added sodium carbonate, the discussers use a saturated carbon dioxide atmosphere.

The 800 C. temperature for the final sulfated residue appears to be necessary to secure the decomposition of aluminum sulfate which takes place at 770 C.[8] The iron sulfate decomposes at a much lower temperature, 480 C.[9]

MESSRS J. B. ROMER,[10] W. W. CERNA[10] AND H. F. HANNUM[10] (*authors' closure by letter*).—The several discussions add considerably to the value of this paper as they have brought out several features.

Mr. Robert T. Sheen advises that after obtaining the total solids he treats the residue with hydrochloric acid, dries and then heats to a dull red heat in order to obtain organic matter. It has been the authors' experience that such a procedure produces considerable error for the reason that this treatment converts all calcium and magnesium alkalinity into the corresponding chlorides and both calcium chloride and magnesium chloride are highly hygroscopic. We have also found that whenever we heat either ferric chloride or magnesium chloride to a dull red heat there is a considerable loss due to volatilization. The authors did not bring out in their paper that they determine organic matter by subtracting the sum of all of the constituents determined by analysis from the total solids obtained by evaporation.

Messrs. Hecht and Whirl point out the fact that they have observed that phosphates tend to increase rather than decrease the weight of the sulfated residue. This means that more complete sulfonation is required in order to avoid this feature. We believe that their comment that the recovery of magnesium sulfate is incomplete is due to a misunderstanding of the authors' paper. We obtain a complete recovery of the magnesium sulfate whereas we may observe a low weight of the sulfated residue due to the fact that some of the SO_3 but none of the MgO may be volatilized from the magnesium sulfate. Such a loss in weight, if it does occur, means a low sodium recovery, but in no way affects the magnesium oxide recovery irrespective of whether it was present in the original sample of water as a sulfate, chloride, or an alkali.

The International Critical Tables do not give any data on the decomposition temperature of magnesium sulfate. Hillebrand and Lundell[11] state that decomposition starts at 1634 F. (890 C.), and becomes energetic at 1782 F. (972 C.). Our experimental data support these statements. Therefore, the heating should be carried out below 1650 F. (900 C.), and preferably at temperatures of about 1600 F. (870 C.).

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[8] *International Critical Tables*, Vol. 1, p. 137 (1930).

[9] Handbook of Chemistry and Physics, Twenty-second Edition, p. 400, Chemical Rubber Publishing Co., Cleveland, Ohio (1937).

[10] Chief Chemist, Assistant Chemist, and Analyst, respectively, The Babcock & Wilcox Co., Barberton, Ohio.

The procedure outlined by Messrs. Hecht and Whirl has the disadvantage that certain salts are hygroscopic and that there is a loss in weight due to volatilization when iron and magnesium chloride are present.

Referring to the comments by Mr. McKinney, it has been the authors' experience that very few waters contain, at the same time, calcium, magnesium, sodium and phosphate. Therefore it is very rare that it will be necessary to go through the many operations for breaking up the mixed R_2O_3 phosphate precipitate.

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[11] W. F. Hillebrand and G. E. F. Lundell, "Applied Inorganic Analysis," p. 545, John Wiley and Sons, Inc., New York City, and Chapman & Hall, London, England (1929).

STATISTICAL METHODS AS AN AID IN REVISING SPECIFICATIONS[1]

By N. C. Wiley[2]

Synopsis

This paper outlines and illustrates several variations necessitated by the available data in applying a method for revising quantitative limits given in specifications for the properties of materials. Only methods described in the widely available standards of the Society are used. The data obtained add to the knowledge of the material and property involved; the relative position of sources of supply indicates the portion of the competition which is on product made and tested under the same essential conditions, and assures revised figures which, it is believed, are as mutually fair to producers and consumers as can reasonably be expected.

Experience in preparing and using specifications applicable to the procurement of materials for the Federal Government has led to the opinion that the numerical values assigned, either as maximums or minimums, to the several quantitative properties of the material really being sought under a specification, is perhaps that portion of the specification least apt to be equitably adjusted between elements of the trade concerned and the consuming activities. Any restrictive or illogical wording used in describing material, workmanship or tests brings forth comment during and after the preparation of the initial issue of a specification, but equally serious defects in the limits assigned to measurable properties are not as readily apparent and are less often the subject of comment. Indeed, the preparing agency and sometimes the trade do not have available adequate data from which to arrive at equitable values at the time of preparing a new specification. A specification for a material not heretofore covered is of necessity based on tests of a few submitted samples or, in some cases, entirely on recommendations. Obviously, in either case, the degree to which the value used as a starting point approximates the true average of all satisfactory material is not definitely known. All that is fairly certain is that there is perhaps some difference, which may be plus or minus, between the assumed and the true average. Specified minimums and maximums should be and usually are set respectively below or above this assumed average, and here the second error may occur. The personnel preparing a new specification can seldom deduce the exact amount to set a limiting value below or above a value obtained from testing one or two submitted samples. If too little or no allowance is made for inevitable variations, it is apparent that much material made and tested within unavoidable limits of chance variation will be considered rejectable. If too liberal an allowance for variations is made, the situation is more serious, for the matter is not apparent, and goes on undetected by the Government until gradually a product of a quality lower than that originally desired can and does monopolize the competitive business essential to Government procurement.

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[1] Opinions or assertions contained herein are those of the author, and are not to be construed as official or reflecting the views of the Navy Dept. or the naval service at large.

[2] Naval Architect, Bureau of Construction and Repair, Navy Dept., Washington, D. C.

The questions with which this paper is concerned then are "can errors in measurable limits inherent in new specifications be rectified on subsequent revisions?," and "if so, how?" A tentative affirmative to the first question is followed by suggestions as to possible methods of rectification.

The Society has been concerned for at least eight years with the application of statistical methods to the interpretation of data. The development of these methods is distinctly a matter for statisticians. Their use, however, has now been made entirely practicable for engineers concerned with material specifications. The "Manual on Presentation of Data",[3] sponsored by the Society's Committee E-1 on Methods of Testing, explains clearly and simply the derivation of and information available from a few simple functions of any given set of *n* numbers and explains the importance of using efficient functions to obtain the useful information available in data. Supplements A and B to this manual set forth in an equally clear and simple manner specific applications of these functions both by rule and example. In what follows, it has not been found necessary to draw extensively on the abundance of literature outside of this manual and explanations and references are purposely largely confined thereto.

The literature on the subject indicates that the application of statistical methods to the properties of materials has been largely to serve one of two purposes:

(*a*) To indicate whether test data, as covered by a report, were obtained under controlled, or essentially homogeneous conditions, in order that an estimate may be made of the "goodness" of the data as a whole, and specific attention directed to any portions of the data for which assignable causes of variation should be sought. The problem of Government specification writing is only indirectly concerned with this application of statistics. However, in passing, the author wishes to mention that statistical treatment of extensive data on cordage, as manufactured by the Navy, has lent confidence in its probable usefulness in other fields.

(*b*) To set up an average and a statistically determined limit about that average as a mutually agreed upon basis of acceptance and rejection between producer and consumer. This is a form of specification with which the author has not had experience. It appears well adapted to cases where a consumer is continuously, for a period of a year or more, taking a fairly constant daily proportion of a plant's output of a certain product. It is not considered, however, to be well adapted to procurement under the usual Government contract, even though the total value of the contract be large. In the first place, any such set-up would have to be described in detail in the invitation to bidders, and could not be made the subject of negotiation after award. Secondly, the low unmodified bidder may be and frequently is one without previous experience with statistical methods of production control, or with the particular specification under which he is about to produce. For example, a jobber or regular selling agent can hardly be expected to maintain or even to have access to production control charts of manufacturers whom he represents. Further, the load on field inspection forces essential to the continuous calculation of statistical functions is out of the question with the usual limitation on funds available for such inspection.

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[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 453 (1933). (Also available as separate publication.)

In view of the considerations immediately above, it appears best to postulate that the numerical requirements of Government specifications will be left as at present; a maximum, a minimum, or both, where necessary. In fact, it has become increasingly the practice to write requirements in the form "970—minimum" in preference to "1000—with a minus tolerance of 3 per cent."

METHOD

In brief, the proposal which follows in detail is to use inspection data, when they become available in sufficient quantity from two or preferably more than two sources of supply, in such a way as to give reasonable assurance that the revised limits will allow acceptance of all material offered that is evidently of the most general commercial grade of the specified quality, to reject the usual material from sources ordinarily producing any lower grades or from usually satisfactory sources when the lot in question contains undesired variations in quality from some unusual cause, known or unknown, greater than should be left to chance, and to insure that the new limits do not, without intent or reason, limit competition to a grade higher than necessary.

The proposed steps in order are:

Step 1. Ascertain for groups (by source) of data on any property, the group averages X and group standard deviations σ, using methods illustrated in Table VI of the A.S.T.M. Manual. In no case is it advantageous to apply statistical methods to data from a source where n is less than 10, and preferably n should be 25 or more.

Step 2. Find the average ($\bar{\bar{X}}$) and the standard deviation ($\bar{\sigma}$) of all data in the groups which are within controlled limits, using the methods given in Supplement B of the Manual.

Step 3. Determine, by the method of Section 4 of Supplement A, the limit of error of $\bar{\bar{X}}$ which might reasonably be present in the data. This possible error becomes less as the amount of available data increases. Table II of this section gives a constant of 1.645 for chances of 9 in 10 that the average of the universe (X') falls within the computed range, and a constant of 2.576 for chances of 99 in 100. These chances become 95 in 100 and 199 in 200 when only a maximum or minimum is of concern, as is usually the case. Examination of data indicates that a constant of 2 is more reasonable than 2.576 in the present application. The corrected average is expressed as X max. or X min.

Step 4. From a function t_1 of $\bar{\sigma}$ for the available data, determine specification limits below or above X max. or X min. The recommended function t_1 is 2.5. From Fig. 14 of the Manual, it will be seen that, for data obtained under controlled conditions, the approximate percentage of total observations lying within the range $\bar{\bar{X}} \pm t_1\bar{\sigma}$ is 98.8 when $t_1 = 2.5$. Again, concern is usually with a maximum or a minimum. Assuming symmetrical distribution about the average, this percentage is then 99.4 in the direction with which the specification is concerned.

In planning test programs, it is advisable, as a study of Supplement B will indicate, to take equal amounts of data from each source. However, the amount of inspection data from a source is dependent on the number and size of contracts or orders which that source has secured, which is rarely equal for two sources. Where n from the least source is over 25 and at least one-half of n from other sources, it somewhat simplifies subsequent operations to take observations from all sources equal to n as available from the least source, discarding the oldest data. For example, if 40 values are available from source A, 50 from source B and 60 from source C, the oldest 10 from source B and the oldest 20 from source C would then not be used.

EXAMPLES

The following illustrative cases discuss applications of the above rules to several typical cases for which available data were used:

Example I—Strength of 3-in. Manila Rope:

Step 1.

SOURCE	SAMPLE SIZE, n	AVERAGE, $\bar{X}$	STANDARD DEVIATION, σ
A	16	10 866	552
B	16	10 288	364
C	16	9 959	248
D	16	10 256	790
E	16	10 900	474

$\bar{\bar{X}} = 10\,454,\ \bar{\sigma} = 486$

Step 2.—The above data can be handled in a manner similar to example B3 of Supplement B.

Control Limits

For $\bar{X}$: $\bar{\bar{X}} \pm A_1\bar{\sigma} = 10{,}454 \pm 0.788\ (486)$
$= 10{,}837$ and $10{,}071$

For σ: $B_3\bar{\sigma}$ and $B_4\bar{\sigma}$
$= (0.443)\ (486)$ and $(1.557)\ (486)$
$= 215$ and 758

Only B lies entirely within these limits, but A and E are close, therefore these three are averaged, giving $\bar{\bar{X}} = 10{,}685$, $\bar{\sigma} = 463$.

Step 3.

$\bar{\bar{X}} - \dfrac{2\bar{\sigma}}{\sqrt{\bar{n} - 3}}$, where $\bar{n} = 3 \times 16 = 48$ gives
X min. $= 10{,}457$

Step 4.

X min. $- 2.5\ \bar{\sigma} = 9390$
The minimum strength at present specified is 9000 lb.

Example II—Soft Rubber for Gaskets, Compression Set in per cent:

Step 1.

SOURCE	SAMPLE SIZE, n	AVERAGE, $\bar{X}$	STANDARD DEVIATION, σ
F	68	19.85	1.90
G	68	19.82	1.34

$\bar{\bar{X}} = 19.835,\ \bar{\sigma} = 1.62$

Step 2.—The above data can be handled as in example B1 of Supplement B. Whereas, in the example above, only minimums were of concern, in this example only maximums are involved.

Control Limits

For $\bar{X}$: $\bar{\bar{X}} \pm \dfrac{3\bar{\sigma}}{\sqrt{n}} = 20.44$ and 19.26

For σ: $\bar{\sigma} \pm \dfrac{3\bar{\sigma}}{\sqrt{2n}} = 2.04$ and 1.20

The above indicates that, in so far as compression set is concerned, the product of sources F and G are statistically in a controlled group.

Step 3.

$\bar{\bar{X}} + \dfrac{2\bar{\sigma}}{\sqrt{\bar{n} - 3}}$, where $\bar{n} = 2 \times 68 = 136$ gives
X max. $= 20.116$

Step 4.

X max. $+ 2.5\bar{\sigma} = 24.166$

The maximum compression set at present specified is 25 per cent.

Example III—Soft Rubber for Gaskets, Hardness, minimum indentation in millimeters:

Step (*1*).—The available data can be handled as in example B2 of Supplement B.

SOURCE	SAMPLE SIZE, n	AVERAGE, $\bar{X}$	STANDARD DEVIATION, σ	VARIANCE, σ^2
F..	31	1.974	0.144	0.013
G..	20	1.999	0.155	0.024
$\bar{n} =$	51			
Weighted average =		1.984		0.0173

For $\bar{X}$: $\bar{X} = 1.984$
For σ: $\bar{\sigma} = \sqrt{0.0173} = 0.132$

Control Limits

Limits for $\bar{X}$: $\bar{\bar{X}} \pm \frac{3\bar{\sigma}}{\sqrt{n}}$

$n = 31$; 2.055 and 1.913
$n = 20$; 2.072 and 1.896

Limits for $\bar{\sigma}$: $\bar{\sigma} \pm \frac{3\bar{\sigma}}{\sqrt{2n}}$

$n = 31$; 0.182 and 0.082
$n = 20$; 0.195 and 0.069

The above indicates that in so far as hardness is concerned, the product of sources F and G are statistically in control. The averages and standard deviations from which control limits were determined took cognizance of two values of n. However, the mere fact that more data were available from source F than from source G is not a good reason for weighing the average after presence or lack of control has been determined. It appears probable that source F had a larger contract or more contracts. Although not a proper inference in this example, for many properties of materials it might be inferred that the source from which the most inspection data were available had a bidding advantage due to inferior quality. Consequently, in steps 3 and 4, the statistical data of this example is averaged, but not weighted.

Step 3.

The new $\bar{\bar{X}}$ is 1.9865, the new $\bar{\sigma}$ is 0.1345, n is 51.

$\bar{\bar{X}} - \frac{2\bar{\sigma}}{\sqrt{n-3}}$ gives $\bar{\bar{X}}$ min. $= 1.9477$

Step 4.

$\bar{X}$ min. $- 2.5\,\bar{\sigma} = 1.6115$

The present minimum for the thicknesses covered by the data is 1.85-mm. indentation. The question before those responsible for this specification is then whether the material having a statistical limit of 1.6115 mm. is satisfactory, in which case the specification should be revised, or whether a limit of 1.85 mm. is so fundamentally important in the application of the material that manufacturers must increase their average or decrease their deviation to meet the present limit or some new limit between 1.6115 and 1.85.

Example IV—Soft Rubber for Gaskets, per cent of original tensile strength after aging at 90 C. for 46 hr.:

Step 1.

SOURCE	SAMPLE SIZE, n	AVERAGE, $\bar{X}$	STANDARD DEVIATION, σ
F..........	73	91.2	2.64
G..........	73	101.3	9.58
	$\bar{\bar{X}} =$	96.25,	$\bar{\sigma} =$ 6.11

Step 2.—As for example II, the above data can be handled as in example B1 of Supplement B, except that, where example II was only concerned with maximums, the specification as to aging states that the "change" in strength shall not exceed a limit. This wording involves maximums and minimums.

Control Limits

Limits for $\bar{X}$: $\bar{\bar{X}} \pm \frac{3\bar{\sigma}}{\sqrt{n}} = 98.34$ and 94.11

Limits for σ: $\bar{\sigma} \pm \frac{3\bar{\sigma}}{\sqrt{2n}} = 7.63$ and 4.59

Step 3.—Here is obviously a new problem, as sources F and G are in separate groups statistically, both as to average and deviations. If limits are determined from $\bar{\bar{X}}$ and $\bar{\sigma}$, they will be purely hypothetical, and material from both sources F and G will be rejectable. If a third source had sufficient data available, two out of three might be in control, and the data from the third source discarded in steps 3 and 4. However, the logical procedure with the present data appears to be to determine limits for sources F and G separately.

For source F

$$\bar{X} \pm \frac{2\sigma}{\sqrt{n-3}}, \text{ where } n = 73$$

$$\bar{X} \text{ max.} = 91.8, X \text{ min.} = 90.6$$

For source G

$$X \pm \frac{2\sigma}{\sqrt{n-3}}, \text{ where } X\text{max.} = 103.6, X\text{min.} = 99.0$$

Step 4.—Another consideration not covered by previous examples is introduced in this step. The general specification applicable to testing rubber states that for tensile strength determinations, the average of the results on four specimens shall be taken from a sample. The 73 determinations available from sources F and G were single observations, but a rejection would of necessity be based on the average of 4 observations. To allow for this, instead of using $2.5\bar{\sigma}$ as in step 4 of previous examples, $\frac{2.5\sigma}{\sqrt{m}}$, where $m = 4$, is used here.

For source F

$$\bar{X} \text{ max.} + \frac{2.5\sigma}{\sqrt{m}} = 95.1, \ \bar{X} \text{ min.} - \frac{2.5\sigma}{\sqrt{m}} = 87.3$$

For source G

$$\bar{X} \text{ max.} - \frac{2.5\sigma}{\sqrt{m}} = 115.6, \ \bar{X} \text{ min.} - \frac{2.5\sigma}{\sqrt{m}} = 87.0$$

From the above, it is seen that limits set for source G include all controlled product from source F. Accordingly, in so far as the above data are concerned, the specified requirement that aging shall not cause a change in tensile strength of more than 20 per cent could be changed to require that the decrease in tensile strength shall be not more than 13 per cent nor the increase be more than 15.6 per cent.

Before leaving the soft rubber gaskets, a new problem brought up by the aging data should be mentioned. If sources F and G are in separate statistical groups as to one property, should they not be treated separately as to all properties? This would seem to depend on the inter-dependence of the properties.

Example V—Yield Point of Structural Steel for Ships:

Step 1.

SOURCE	SAMPLE SIZE, n	AVERAGE, $\bar{X}$	STANDARD DEVIATION, σ
H.........	51	38 733	765
I..........	115	40 907	3050
J..........	24	39 102	1010
K..........	65	38 607	2179
L..........	10	45 948	2910
M..........	19	39 820	2510
N..........	34	41 346	2990
$\bar{\bar{X}}$ weighted		40 092, $\bar{\sigma}$ =	2371

Step 2.—This step is handled as in example III.

Control Limits

SOURCE	LIMITS FOR $\bar{X}$		LIMITS FOR σ	
H....	41 088	39 096	3075	1667
I.....	40 756	39 428	2840	1902
J.....	41 544	38 640	3397	1345
K....	40 974	39 210	2994	1748
L.....	42 343	37 841	3962	780
M....	41 721	38 461	3526	1216
N....	41 382	38 802	3233	1509

From the above, it is seen that sources J, M and N are statistically in a controlled group as to $\bar{X}$. The relative extent to which σ for source J is below the lower limit is not considered to warrant discarding source J data. Using these three sources gives $\bar{X} = 40{,}089$, $\bar{\sigma} = 2170$, $\bar{n} = 77$.

Step 3.

$$\bar{\bar{X}} - \frac{2\bar{\sigma}}{\sqrt{n-3}} = 39{,}584 = \bar{X} \text{ min.}$$

Step 4.—The consideration as to a group of observations forming the rejection basis applies here, except that m equals 2 instead of 4 as in example IV.

$$\bar{X} \text{ min.} - \frac{2.5\bar{\sigma}}{\sqrt{m}} = 35{,}691$$

This limit is higher than the specifications in effect at the time this analysis was made. As a matter of interest, specifications revised since this analysis was made, but not based thereon, give minimum yield points varying from 37,000 lb. per sq. in. for thicknesses under ½ in. to 33,000 lb. per sq. in. for thicknesses over $\frac{5}{8}$ in. The data used in the above analysis cover this entire range. A separate analysis indicates the desirability of the revision made and accounts for some of the averages and deviations in the above data. For example, source L was lighter weight material than from other sources, accounting for the high $\bar{X}$, source I covered a wide range of weights, accounting in part for the high σ.

As a practical point, saving possible future repetition of work, it is recommended that the summations of observations and of their squares used in step 1 be retained in file, in order that further data may be conveniently added thereto. For example, the three properties of rubber are based on data from two sources. Data from a third source are obviously highly desirable, before a revision of specified figures is put into effect. When these data are available, it will only be necessary to add the sums to those for sources F and G to obtain new control limits.

DISCUSSION OF RESULTS

It is evident from the treatment of the foregoing data that cases may be expected to arise where judgment must be exercised, and that therefore statistical methods are an aid to, but not a substitute for, practical engineering judgment.

With the aid of the above examples, two questions which may be raised with respect to steps 3 and 4 are now considered further. First, why leave a possibility of rejection by using the constant 2 in lieu of 2.576 in step 3, and by using 2.5 in lieu of 3 in step 4? The reason is that the adopted functions of σ give results which are reasonable when applied to data at hand, whereas the extreme functions, while more nearly approaching theoretical limits, give values so far beyond the body of available data as to be unacceptable to the Government, and their adoption might lead those responsible for the quality of material to the conclusion that statistical methods are not of as much value as a practical guess. Figure 1 shows graphically the data used in examples I, II and III. The dotted limit lines, using the above pair of larger functions, are obviously far beyond the range of the body of observations, and the full limit lines, using the adopted functions, show a liberal margin. The four observations beyond the limits for compression set are obviously not the result of the same essential system of causes as the body of the observations. The unshaded portion of the graph for manila rope indicates observations on the data from sources C and D, which data were discarded in step 1 of example I. The fact that these additions lie within the derived limit is indicative of the liberality resulting from its use. As was pointed out in that portion of this paper in which the derivation of the recommended steps and functions was outlined, the chances are nearly 98 in 100 that $\bar{X}$ min. is lower or $\bar{X}$ max. is higher

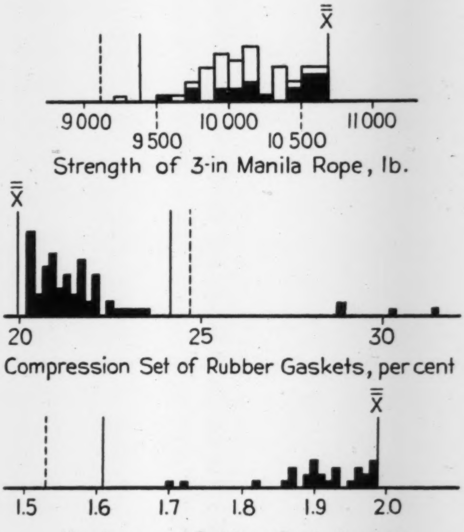

FIG. 1.—Data Used in Examples I, II and III.

than the true average, and more than 99 in 100 observations may be expected to lie between this remotely possible value of the average and the final specification limit. As these two rejections of chance are superimposed, adequate protection is certainly given the producer.

The second question which may be raised is as to why step 3 is not discarded and $3\bar{\sigma}$ used in lieu of $2.5\ \bar{\sigma}$ in what has been given as step 4. This would slightly simplify the procedure, but is not considered to result in as accurate an estimate as that adopted. The average is fully as uncertain a quantity as the deviation in analyzing data. An example is the broad field of textiles and cordage, for which data collected in any given year are, to a considerable extent, based on fiber taken from the crop of the preceding year. Variations in weather conditions from year to year may affect average strength more than it affects the standard deviation of strength observations. The elimination of step 3 would remove a valuable relationship between the certainty of the average and the amount of data available. Where $\bar{n}$, as used in step 3, is 20 or more, the procedure recommended herein will give limits closer to the average than limits obtained by eliminating step 3 and using $\bar{\bar{X}} - 3\bar{\sigma}$ in lieu of $\bar{\bar{X}} - 2.5\ \bar{\sigma}$ in the last step, but data based on a total of less than 20 observations is inadequate for proper statistical determination of specification limits. As to the work saved by this modification, it should be borne in mind that no one person undertakes the revision of a specification at frequent intervals, and that the major portion of the procedure herein advocated has been completed when $\bar{\bar{X}}$ and $\bar{\sigma}$ have been determined. The limits resulting from the three functions just discussed, as applied to the five examples above, are tabulated in Table I. These limits have been rounded off to three significant figures as they probably would be in a specification.

TABLE I.

Example	$\bar{n}$	$\bar{\bar{X}}$	$\bar{\bar{X}} \pm \frac{2\bar{\sigma}}{\sqrt{\bar{n}-3}} \pm \frac{2.5\bar{\sigma}}{\sqrt{m}}$	$\bar{\bar{X}} \pm \frac{2.576\bar{\sigma}}{\sqrt{\bar{n}-3}} \pm \frac{3\bar{\sigma}}{\sqrt{m}}$	$\bar{\bar{X}} \pm \frac{3\bar{\sigma}}{\sqrt{m}}$
I min......	48	10 700	9 390	9 120	9 300
II max.....	136	19.8	24.2	24.7	24.7
III min......	51	1.99	1.61	1.53	1.58
IV min......	73	101	87.0	86.6	86.9
IV max.....	73	101	116	116	116
V min......	77	40 100	35 700	35 400	35 500

TABLE II.

$\bar{n}$	t_1	$\bar{n}$	t_1	$\bar{n}$	t_1
40	2.829	80	2.728	120	2.685
45	2.809	85	2.721	125	2.681
50	2.792	90	2.714	130	2.677
55	2.777	95	2.709	135	2.674
60	2.765	100	2.703	140	2.671
65	2.754	105	2.698	145	2.668
70	2.744	110	2.693	150	2.665
75	2.736	115	2.689	155	2.662

In order to clarify the separate effects of allowing in step 3 for possible errors in the average and in step 4 for variations about the final average to be expected in a controlled product, these steps were treated separately in the examples given. However, it is evident that, when m equals 1, steps 3 and 4 can be combined and expressed as $\bar{\bar{X}} + t_1$, where $t_1 = \frac{2}{\sqrt{\bar{n} - 3}} + 2.5$, $\bar{n}$ being the total number of observations in the groups in control. Table II gives t_1 for a range of values of $\bar{n}$.

DISCUSSION

Mr. W. P. Roop[1] (*presented in written form*).—I should like to ask the author several questions:

1. Assuming all desired data available, where on the frequency curve should the specified values intervene? Manufacturers prefer no rejections; inspectors feel they must make at least a few. Perhaps there is an optimum, but there are economic factors in finding where it is; the marginal producer will naturally have more rejections than the best producer. But should even the best producer have no rejections at all?

2. Where should the line be drawn between the two aphorisms: "The best is none too good" and "Good enough is best"? The author bases his analysis entirely on the first of these, and inquires only what is obtainable, not what is needed.

3. What does the author mean by "errors"? I gather that he visualizes a desirable value which may be known or unknown. If known, the problem in revision is to reduce "error" by adjusting specified values to the desirable value within a tolerance fair to all concerned. In principle his detailed statistical methods have the purpose of determining the values and tolerances to be specified by a process of analysis of actual values obtained in previous tests. From this point of view the "errors" which he wishes to rectify are essentially administrative in character, since the effect of such errors is felt mainly in restriction of sources of supply, or in excessive or inadequate profit to the suppliers. I do not wish to question the adequacy of his analytical procedure; and the formal adoption of some definite procedure in this matter is most commendable.

4. But what about the more usual case where the desirable value is unknown? For the present purpose it would be sufficient to point out that this question is not discussed by the author. Naturally he is quite right in restricting himself to a definite field of inquiry, but it is well to be quite clear as to the limits of application of the considerations he advances.

5. Questions as to desirable, as distinguished from obtainable, values partake more of an engineering nature. Is it possible to say that the best manila rope is the strongest, the best gasket is the softest, or the best steel that with the highest yield point? Of course not. Even aside from matters of cost, there are always limits in the other direction too. So that when the author prefers, as he says, a minimum, rather than a normal value with tolerance, he must combine with that a minimum in some other opposed property, whether this be tacit or expressed. As an anomaly, I may cite the minimum and maximum values specified for ultimate strength of high tensile steel: why not a minimum ductility in place of the maximum strength? And particularly how shall we discover, by statistical analysis or otherwise, how much ductility is necessary?

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MR. H. F. DODGE.[2]—This paper is very timely and worthy of deliberate consideration. It provides a specific procedure for the selection and use of uniform data in establishing maximum and minimum limits in specifications. As engineers we know of course that purely statistical techniques are applicable only under rather idealized conditions and that we must always fall back on the use of judgment when such conditions do not obtain. For example, a committee is often placed in the position of rendering a decision in setting specification limits based on qualitative information that is of greater weight than the quantitative information at hand. I have in mind the case where one supplier, who is known to be consistently reliable and whose data are available in large quantities, may be found by the statistical control test to be outside of "control limits" established from the combined results of several suppliers. In such an instance, it may further be known that this supplier's product is eminently satisfactory for the intended service. With this knowledge at hand, the committee may wish to set a limit which will include the reliable supplier even though the proposed control test suggests his exclusion. The last section of the paper which states that "statistical methods are an aid to, but not a substitute for, practical engineering judgment" appears to recognize this type of situation and thus provides the latitude needed by a committee in evaluating the data at hand. Bearing this reservation in mind, it is believed that the proposed method can be used to considerable advantage by some of our standing committees.

MR. N. C. WILEY[3] (*author's closure by letter*).—Replying to the first paragraph of Mr. Roop's discussion, the author had concluded that limits as determined from the function of σ as given in Table II would approximate an optimum. The question of whether the best producer will have any rejections under limits determined as suggested depends on how much "the best" that producer is. If one producer has a distinct margin above all others, a limit causing a serious probability of rejection of his product so limits the balance of the producer group as to result in practically eliminating competition.

Mr. Roop concludes that the author bases his analysis entirely on the aphorism that "The best is none too good." The following quotations from the outline of the proposed method are pertinent in this connection, " . . . to give reasonable assurance that the revised limits will allow acceptance of all material offered that is evidently of the most general commercial grade," and further on " . . . to insure that the new limits do not, without intent or reason, limit competition to a grade higher than necessary."

Mr. Roop's reference to the use of the word "errors" evidently refers to the question "Can errors in measurable limits inherent in new specifications be rectified on subsequent revisions?" With respect to the statement that the effect of such "errors" is felt mainly in restriction of sources of supply, the paper emphasizes the fact that "errors" having such an effect are brought to light by those sources which are eliminated or caused difficulty. As the paper points out, it is the "error" which results in insufficient restrictions in source which is most apt to continue indefinitely unless detected by statistical analysis.

It is not clear to the author why a

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minimum must be combined with a minimum in some other opposed property if a normal value, with a minus tolerance, need not be so opposed. It appears merely to be two ways of expressing the same limits, the use of a minimum involving the least arithmetic. The mentioned relation of minimum ductility and maximum strength, if involving statistics, introduces correlation, a statistical application admittedly not covered in the paper or, for that matter, in the Society's "Manual on Presentation of Data."

The author is pleased beyond anticipation that Mr. Dodge finds the paper timely and worth deliberate consideration. Mr. Dodge and Mr. Roop both indicate the necessity of recognizing that the procedure has limitations, and is by no means a "cure-all." However, with an analysis as outlined at hand, it is considered that either engineering or administrative judgement will be based on a more complete and accurate picture than would otherwise be available. It might be considered that the results of the statistical analysis completes the data or that it provides one of the considerations upon which the ultimate decision is based.

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